

THE DESIGN AND WORKING
OF AMMONIA STILLS

THE DESIGN AND WORKING OF AMMONIA STILLS

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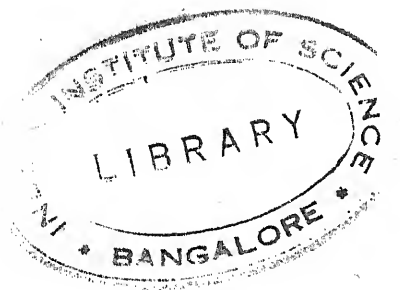
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FOREWORD*

BY

DR. CHARLES C. CARPENTER, C.B.E., M.I.C.E.,
PRESIDENT OF THE SOUTH METROPOLITAN GAS COMPANY

AMONG the direction posts standing up out of the débris of the Great War, few are of greater importance to the Nation than those pointing the need of a developed Chemical Industry for ensuring its security.

At all centres of learning a more or less widespread knowledge exists of those fundamentals of Chemical Science, the variations on which can be produced or directed as necessity may require. If the note-books of the workers thereat cannot be classed as "books of recipes," to be drawn upon in times of trouble, they constitute, at any rate, records of combinations and reactions, together with an ever-widening understanding of their properties and potentialities.

But to put these stores of knowledge to practical large scale use depends less on the inspiration of experiment than on the application of experience, both phases of development conforming, of course, to the basic principles underlying them. The one branch becomes the subject of abstracts made by the various chemical societies, and their library shelves form a store house of invaluable material which can be utilised at short notice. The other is for the most part buried in the process records and works drawings of the chemical manufacturers, by whom they are generally jealously guarded.

It is for this reason that the book Mr. Parrish has prepared with such thoroughness is likely to be so valuable to those interested in the technology of his subject. We do not mind the ruggedness of the writing nor the absence of polish in the phrasing. We turn to his pages, not for inspiration, nor for visions, but for facts and for figures, and we are provided with the very things we seek. We may be sure that when Mr. Parrish gives us a working drawing of a still we can send it straight away to the manufacturer, certain that when constructed and erected it will function in the

** The author desires to express his deep sense of indebtedness to Dr. Carpenter for having written the Foreword to this volume.—P. P.*

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manner expected of it. Collaboration between those responsible for the design of large scale chemical plant can only be a war-time proposition. Meanwhile the industry will continue to owe much to the unselfish labours of workers of the type of Mr. Parrish. He has placed, through his book, his wide knowledge at the service of all seeking information upon the subject of which he treats, and has given particulars of yields and costs which must prove of great utility to all engaged in the conduct of like operations.

CHARLES CARPENTER.

July 1924.

PREFACE

It is estimated that 300,000 tons of sulphate of ammonia are annually produced in Great Britain by the direct distillation of the ammoniacal liquors arising from gas, coke oven, shale distillation, and blast furnace works. The cost of production of steam is appreciably higher to-day than it was a decade ago. This has had an influence on the production costs of sulphate of ammonia, in which steam represents 15 to 20 per cent. Yet in no volume or treatise can anything really satisfactory or fundamental be found concerning the design of ammonia stills in its relation to steam consumption.

That there are many unexplored fields which lie within the reach of the gas and coking industry is undoubted. Of these, not the least significant is the one relating to the use of steam in the distillation of gas and coke oven liquors.

By a modification of existing plant a substantial saving in the cost of the manufacture of sulphate of ammonia can be effected.

This book attempts to deal with the fundamental principles underlying the design of stills, preheaters, condensers, dephlegmators, and coolers, etc., with special reference to economy of steam, and the utilisation of potential heat. It foreshadows a rearrangement of plant for the manufacture of sulphate of ammonia, and it aims at conveying new conceptions which should be of value.

In addition, the working and control of stills as affecting many operations are dealt with in a practical manner, and in a way which it is hoped will appeal not alone to chemists, engineers, and technologists, but to chemical works operatives.

The pecuniary aspect has not been overlooked, and it is believed that many commercial men will welcome the chapter dealing with the considerations governing the price of gas liquor, as also the financial statements furnished in Chapters VIII. and XI.

In attempting to make the book of real service to the gas, coke oven, and chemical industries, the author has taken advantage of the generous help of many chemical and gas engineering firms, and he desires to acknowledge fully the assistance so readily given in furnishing blocks, drawings, and other useful data. The author has made use of the Chief Alkali Inspector's Reports, the proceedings of technical societies, and articles appearing in the technical press. In such cases due acknowledgment has been made in the text.

Moreover, his thanks are due to his colleague, Mr. O. W. Weight, who has

critically perused most of the typescript, and from whom many valuable suggestions have been received. Mr. F. C. Snelling has undertaken the task of reading the volume, and of checking the calculations and heat balances, and the assistance which he has rendered is highly appreciated, as indeed is that of Messrs. C. E. Parr and C. H. E. Maggs.

It is impossible to prepare a book of this character without incorporating the ideas and principles which are intuitively acquired as a result of association with the officials and staff of the Company with which one is connected. Whatever merit this book may possess as an endeavour to reach fundamental principles, it is certain that this is in no small degree attributable to Dr. Chas. C. Carpenter, M.I.C.E., and Mr. E. V. Evans, F.I.C. (Chief Chemist and Products Manager of the South Metropolitan Gas Company), for whose kindly help and encouragement the author wishes to record his appreciation, as indeed his thanks for permission to publish this volume.

P. PARRISH.

BLACKHEATH, S.E.

June 1924.



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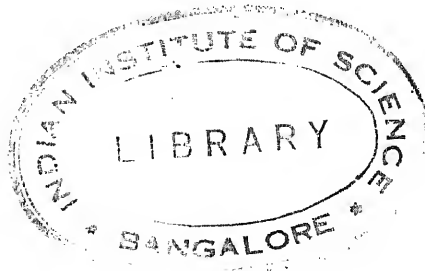
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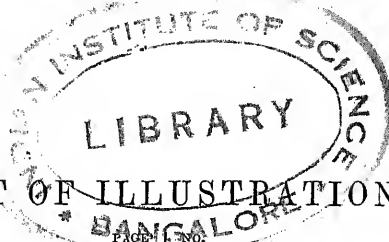
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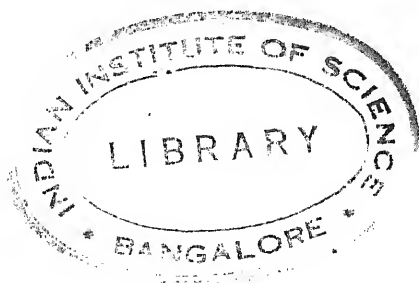
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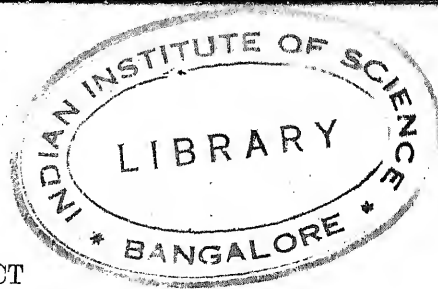
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CHAPTER I

A GENERAL SURVEY OF THE SUBJECT

INTRODUCTORY

THAT a need exists for a manual dealing with the design and working of ammonia stills is undoubted. Singularly little published matter is to be found respecting the design of plant in which distillation operations are effected. Why this is so is not far to seek. Many processes have been handed down by plant producers in the chemical engineering industry undeveloped, or developed to such an extent as to be of practical utility. Distillation plant is no exception. But the plant has invariably preceded the determination of the physico-chemical data which are calculated to aid design, and which, presumably, would ensure a more efficient unit of plant. In other words, design in the past has been largely empirical, and engineers have not been in a position, or if in position have been disinclined, to give details of the considerations leading to the adoption of the characteristics of the plant.

One would hardly expect distillation apparatus evolved under the conditions indicated to reach the acme of perfection. It is certain that questions of heat transmission, and the economics of steam consumption in their relationship to the physico-chemical principles involved, have not been the dominant considerations of the design. Not infrequently, purely mechanical considerations have been the factors largely governing the evolution of stills and accessory plant in the past. The almost complete absence of any definite attempt to correlate or co-ordinate the theorems of the laboratory to actual still design is striking. Much excellent matter has been published on the subject of fractional distillation. Indeed, Young's book on this subject may be regarded as a model of its kind. But it is essentially one dealing with the aspects of fractional distillation as viewed from the chemical laboratory. Young does not sensibly concern himself with the application of the laboratory processes to plant.

Any initial attempt at co-ordination must be largely tentative, but a measure of its success will be gauged by the extent to which the present darkness is permeated by the illuminating rays of such co-ordinating effort. So insistent has the need for purity become during the last few years that many manufacturers have been perforce compelled to refine their products in order to market them successfully. Fractional distillation has often offered the most readily available and most economical means to this end. Thus there has been a distinct impetus to creative development, and improvements in still design and accessory plant must sooner or later inevitably result.

To cover the whole gamut of distillation operations would be impossible in the present volume. The author's intention to limit the present manual to ammonia stills and accessory plant merely arises from a desire to deal with the subject in some detail, rather than attempt to cover a larger field in a general manner.

From communications which have been received from several sources, it has

been made increasingly evident by chemists, coke oven managers, gas engineers, and chemical manufacturers that a book dealing with the various ammonia distillation processes from the points of view of design and working would be appreciated.

Four considerations of significance are ever dominant in connection with ammonia distillation processes to-day. These are (1) increased productive capacity consistent with efficient removal of the ammonia; (2) economy of steam consumption; (3) efficient utilisation of available heat; and (4) the disposal of the waste liquor.

Without doubt, the increased cost of labour and the competition, both actual and prospective, of other sources of ammonia account for the first consideration. The second and third aspects are largely due to the increased cost of fuel—both high class and low grade. The fourth consideration is the result of greater activity on the part of county councils and river boards in preventing pollution of rivers and streams by waste liquor of a noxious character.

To ensure greater productive capacity of the still with the maintenance of a satisfactory waste liquor, and with economy of steam, is not a simple proposition.

By a modification of the design of stills an economy of steam can be effected; and certainly efforts must be made in this direction. But so far as concerns ammonia stills used in the manufacture of sulphate of ammonia, the greatest economy is likely to accrue from a better utilisation of the available heat of the process. The existing arrangement of plant does not provide for anything approaching complete conservation of the heat rendered serviceable. An attempt will be made to show the directions in which improved working results can be secured in this connection.

Apart from the foregoing aspects, the more efficient generation of steam from low-grade fuel is a problem which will materially assist in fuel economy. Most gasworks and coke ovens have available low-grade fuel, usually in the form of breeze. The value of this material a few decades ago can be readily appreciated from an examination of the following statement of the cost of manufacture of sulphate of ammonia presented by the late Alfred Colson, M.I.C.E., in a presidential address before the Incorporated Institution of Gas Engineers in 1894.

The statement in question is subjoined (see Table 1).

Colson remarked, on the subject of the item for breeze, that “for sulphate of ammonia manufacture steam is raised entirely by breeze dust used under forced draught, and as this material has frequently to be disposed of as refuse at some little expense, sulphate is debited with the cost of carriage only, which is seen to be insignificant.”

In contrasting the above conditions with those existing to-day, when breeze is realising between twelve and fourteen shillings per ton, it will be realised how important the cheap and efficient generation of steam becomes. This is not alone a problem with which this country is faced. It is equally insistent on the Continent, particularly as affecting those works which distil sewage liquors in the production of sulphate of ammonia, and in those factories which regenerate or recover by distillation ammonia in the Solvay ammonia-soda process. The writer therefore offers no apology for assigning a chapter of the present volume to the subject of

A GENERAL SURVEY OF THE SUBJECT

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TABLE I

SULPHATE OF AMMONIA MANUFACTURE FOR THE YEAR 1893

Dr. Ammoniacal liquor distilled, 3,700,096 gallons, 10 oz. strength.			Ammoniacal liquor made, 3,588,096 gallons, 10 oz. strength.			Cr.		
	£	s. d.	Per Ton of Sulphate made.			£	s. d.	
To Stock	4,988	19 7	£ s. d.	By Sulphate of ammonia, 148 tons 14 cwt. .		17,704	17 0	
Salaries	176	5 0	0 2 5-19	Stock		5,035	0 8	
Wages	784	8 1	0 10 9-92					
Repairs and mainten- ance	415	10 7	0 5 8-82					
Fuel	5	16 11	0 0 0-96					
Sulphuric acid, 131 tons 2 cwt.	2,578	6 2	1 15 7-04					
Lime, 136 tons 6 cwt. Bags	129	7 9	0 1 9-43					
Carriage (liquor) . .	165	2 9	0 2 3-35					
Carriage (sulphate) .	99	8 2	0 1 4-46					
Stationery and print- ing	307	14 11	0 4 2-97					
Gas	7	7 11	0 0 1-22					
Water	59	4 3	0 0 9-80					
Depreciation	29	17 1	0 0 4-94					
Interest	463	2 4	0 6 4-70					
Loss on sulphur re- covery	392	2 0	0 5 4-94					
Sundries	35	0 5	0 0 5-80					
	63	9 4	0 0 10-51					
	10,701	3 3	3 18 10-11					
To Balance, being amount realised for the year .	12,038	14 5						
	22,739	17 8						
Amount realised per ton of liquor made (10 oz.), 14s. 9-15d.				Amount realised per ton of coal carbonised, 2s. 3-55d.		22,739	17 8	

steam generation from low-grade fuel, and for indicating directions in which improved results can be attained.

In the matter of gas liquor and boiler feed water preheaters, condensers, dephlegmators, and interchange heaters, no less than stills, to which sufficient reference has already been made, there is need for a reconsideration of the design of such apparatus. It is believed that established practice should be tempered and shaped by the considerations of scientific knowledge and data which have accumulated as a result of careful investigation.

Much of the plant indicated above is not efficiently designed from the point of view of heat transfer. Many types of dephlegmators and condensers in connection with liquor ammonia plants are of an unsuitable character, where the transfer of heat is not aided to any sensible degree by the creation of turbulent motion, and where such factors as viscosity, straight line flow, critical velocity of the liquid and vapour have not been adequately contemplated.

Apart from heat transference in connection with the dephlegmators, etc., of liquor ammonia plants, the removal of the whole of the water in order to produce a dry ammonia gas is a problem fraught with several considerations. As the vapour pressure of ammonia is greater than that of water, by slow and regulated cooling, and the maintenance of the temperature of the steam-ammonia stream slightly below its dew-point, it is possible to reflux the whole of the water necessary to give a dry ammonia gas with a minimum of ammonia. The less the content of ammonia in the reflux, the less heat required for its disengagement, and the less work imposed on the finishing still.

In the decarbonation and desulphurisation of ammoniacal liquors in the production of caustic ammonia there are many interesting problems of design involved, reference to which will be made in detail in the chapter assigned to this particular phase of the subject.

This introductory chapter is merely intended to show the many and complex problems which will need to be treated.

The appropriation of a chapter to the stills and accessory plant used by the ammonia-soda industry should prove apposite in view of the appreciably stronger solutions of ammonia with which these plants invariably deal as contrasted with the ammoniacal liquors arising from gas, coke oven, blast furnace, and shale distillation works.

CHAPTER II

THE PRODUCTION OF STEAM FROM LOW-GRADE FUEL

GENERAL

THERE appears to be no better medium for disengaging ammonia from ammoniacal solutions than steam. Its physical properties undoubtedly favour its use. Those ammonia distillation works which are adjacent to gasworks and coke ovens where large quantities of coal are carbonised or "coked" have immediately at hand a relatively cheap low-grade fuel, when contrasted calorific value for calorific value with the cost of higher grade material.

The production of steam from low-grade fuel becomes at once a matter of fundamental importance to such chemical works. There is still ample opportunity for improvement in the utilisation of this material.

"Coke breeze" is an indefinite term. Some gas engineers describe it as all the material passing a $\frac{3}{8}$ -inch screen. Others would regard such material, or at least that passing a $\frac{1}{4}$ -inch screen, as coke dust. Whether a specification for breeze will ever become practical politics is a matter of doubt. Many attempts have been directed to the adoption of a specification, but so far these efforts have proved unavailing.

CHARACTERISTICS OF BREEZE

The author's experience of coke breeze resulting from gasworks carbonising coal with an ash content of about 9 per cent (dry basis), and where the screening of the coke is efficient, and where care is exercised in quenching, is that an average ash content is 21 per cent, and the average moisture content 10 per cent, giving a calorific value of approximately 10,080 B.Th.U. per pound. Screening tests of such breeze reveal the following characteristics :

TABLE 2

Percentage passing a $\frac{1}{8}$ -inch screen	.	.	.	25.74
" " " $\frac{1}{4}$ -inch "	.	.	.	43.05
" " " $\frac{5}{16}$ -inch "	.	.	.	71.66
" " " $\frac{7}{16}$ -inch "	.	.	.	83.72
Remaining	.	.	.	16.28

If the 25.74 per cent of $\frac{1}{8}$ -inch material was removed by screening, no serious objection could be raised to the combustion of such fuel for steam generation. It is the tendency of the finer material (that passing a $\frac{1}{8}$ -inch screen) to choke the interstices of the otherwise reasonably porous fuel bed, which creates for breeze a certain degree of odium. This tendency to consolidate the mass, thus increasing the resistance and rendering uniform distribution of air at a reasonable pressure next to impossible, is the chief disadvantage which one has to face. It is seen that the fuel

bed is somewhat typical of the sand lute. Although porous, there is no uniform porosity, and such pressures have to be induced by the operation of the steam blasts as invariably lead to an excess of air beyond that theoretically required for the combustion of the carbonaceous matter of the fuel.

FORCED DRAUGHT FURNACES FOR BREEZE COMBUSTION

There are many forced draught furnaces suitable for the combustion of breeze on the market. Those in popular favour are the "Crosthwaite," "Meldrum," "Victoria," "Superheat," "Turbine," and "Wilton."

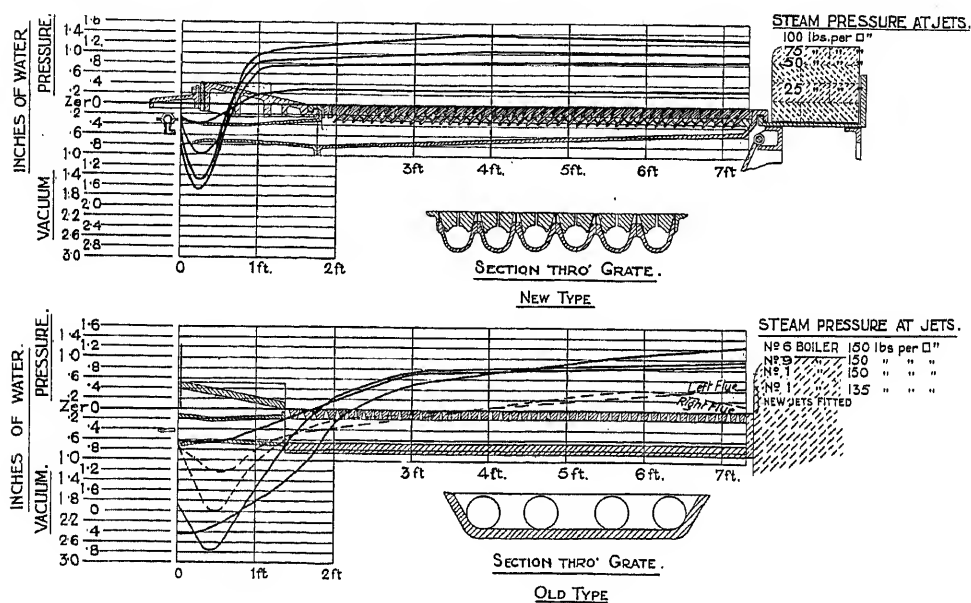


FIG. 1.—GRAPH OF STEAM PRESSURES, ETC.; NEW AND OLD TYPE FURNACE.

Generally speaking, the furnaces named are for the most part constituted of a series of cast-iron air tubes, built in sections. Each section is provided with serrations so arranged that it can form a series of perforations with the abutting section. A blanked end section is provided for each furnace, so that the air for the combustion of the fuel passes through sections, or perforations formed by the various sections when assembled.

The air for combustion is delivered through suitable jets, or nozzles, the orifices of which vary with different makes. In connection with the "Crosthwaite" furnace a nozzle having a $\frac{1}{8}$ -inch diameter orifice is used. With the "Turbine" the diameter of the nozzle is $\frac{1}{16}$ inch.

Recent improvements in the design of furnaces for the combustion of breeze have been directed to the number and size of the holes for the admission of air, with

minimum dust percolation, to the blast pressure, as well as to the production of a supply of secondary air.

That there was room for improvement as regards the distribution of air is demonstrated by an examination of fig. 1, showing how the pressure varies throughout the grate when contrasting a new and an old type of furnace. Fig. 2 shows the "Turbine" patent forced draught furnace, concerning the mechanical construction of which it is not necessary to enter into great detail, as the features are well illustrated in fig. 3. The principle of the furnace is based upon the impulse turbine, the

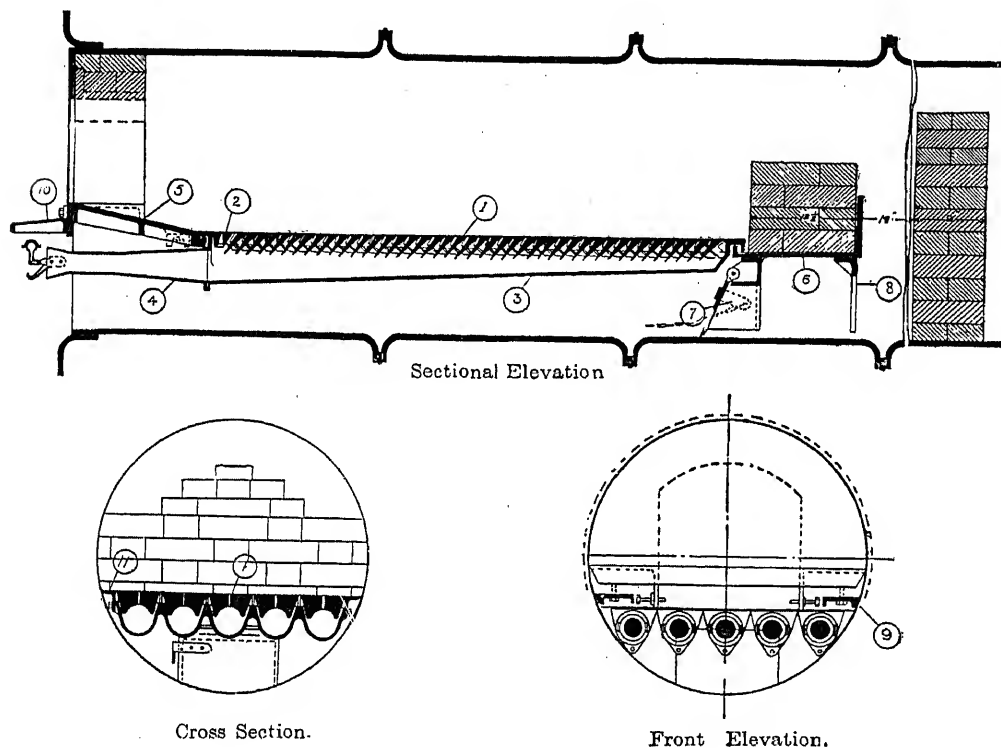


FIG. 2.—SECTION OF TURBINE FIRE-GRATE.

air trough corresponding to the nozzle, and the fire-bars to the blade of the turbine. The air is forced between the bars, and passes into the furnace in the form of fine air sprays. Due to the proportioning of the air trough, and the shape of the bars, equality of air distribution is effected. The steam jets used are exceedingly small, being only $\frac{1}{16}$ -inch diameter, as has already been indicated. The consumption of steam at the jets is said to be very low, and this is one of the important claims in respect of this type of furnace.

Fig. 4 represents a section showing the grate and valvular dead plate as supplied by Messrs. Meldrums, Ltd., of Manchester. The dead plate valve automatically

opens on closing the furnace door, and admits the introduction of the necessary secondary air under positive pressure for whatever period is deemed necessary. By

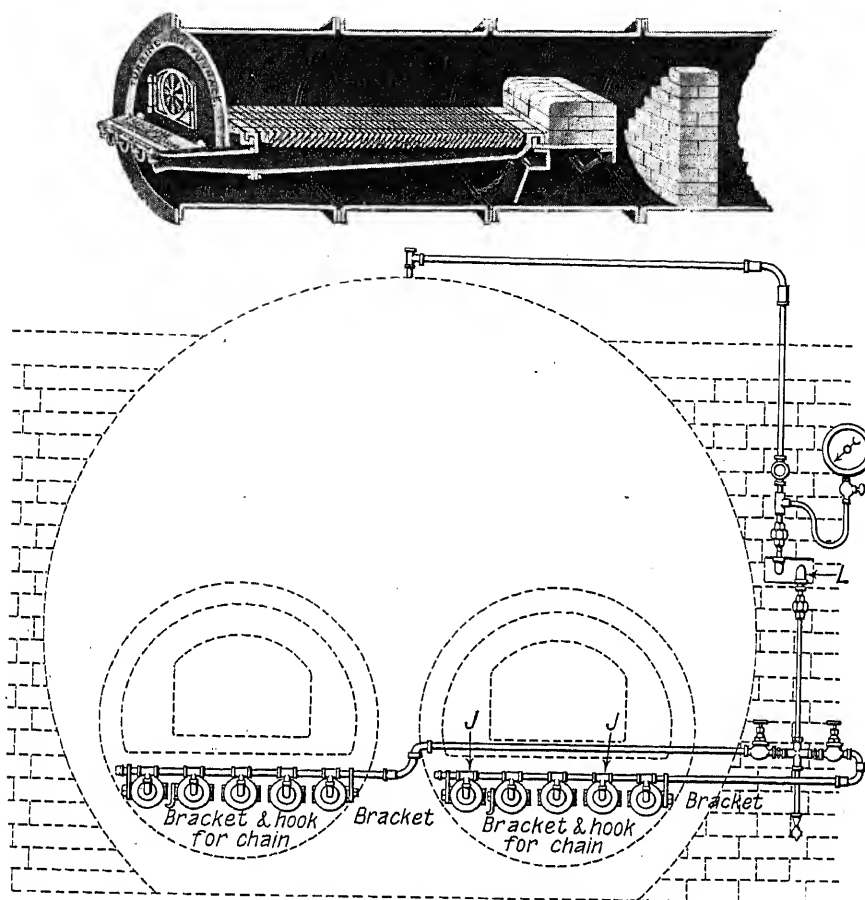


FIG. 3.—FIRE-GRATE AND VIEW OF TURBINE BOILER.

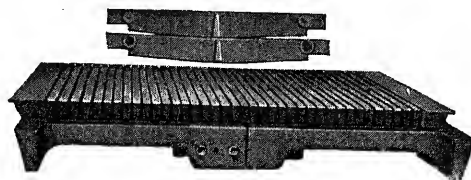


FIG. 4.—MELDRUM'S FIRE-BARS AND GRATE.

a slight side knock of the lever on the furnace door, the closing of the valve can be effected.

Fig. 5 gives details of a recently designed and tested new form of grate bar,

specially adapted for low-grade fuel. It will be seen that vertical or inclined air spaces have been dispensed with, and the draught is exerted horizontally, preventing unburnt fuel dropping through, as alike avoiding undue and upward draught to lift the fuel. Fig. 6 represents the Meldrum forced draught furnace.

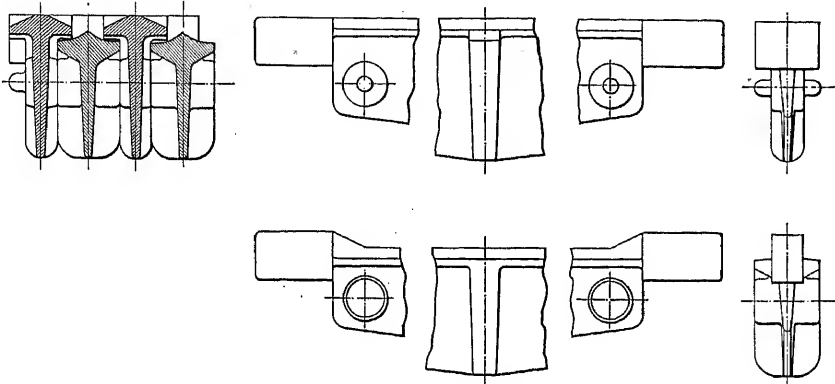


FIG. 5.—MELDRUM'S FIRE-BARS FOR COMBUSTION OF COKE BREEZE.

Consideration of fig. 7, which represents a Crosthwaite bar, and also fig. 12, showing a section of a fire-grate fitted with a Crosthwaite forced draught furnace, will show that the primary air supply is a function of the pressure at which the steam

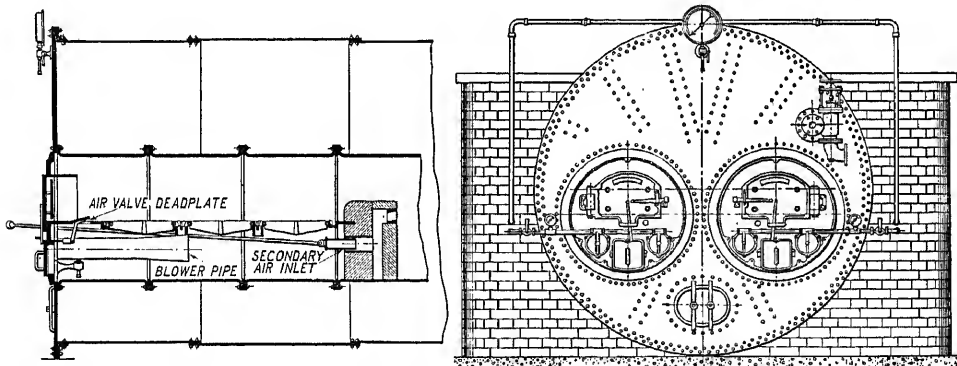


FIG. 6.—MELDRUM'S PATENT FORCED DRAUGHT FURNACE.

blasts are operated, and the resistance caused by the fuel bed. The initial pressure created by forcing air through the fuel bed is counterbalanced by the suction of the chimney, and the point of equilibrium is usually above the fuel bed. The secondary air supply is determined by the chimney draught, and the extent to which the secondary air ports are controlled by the adjustable slides shown. The latter provision is one which the author has found it necessary to adopt in order to afford better control of the secondary air.

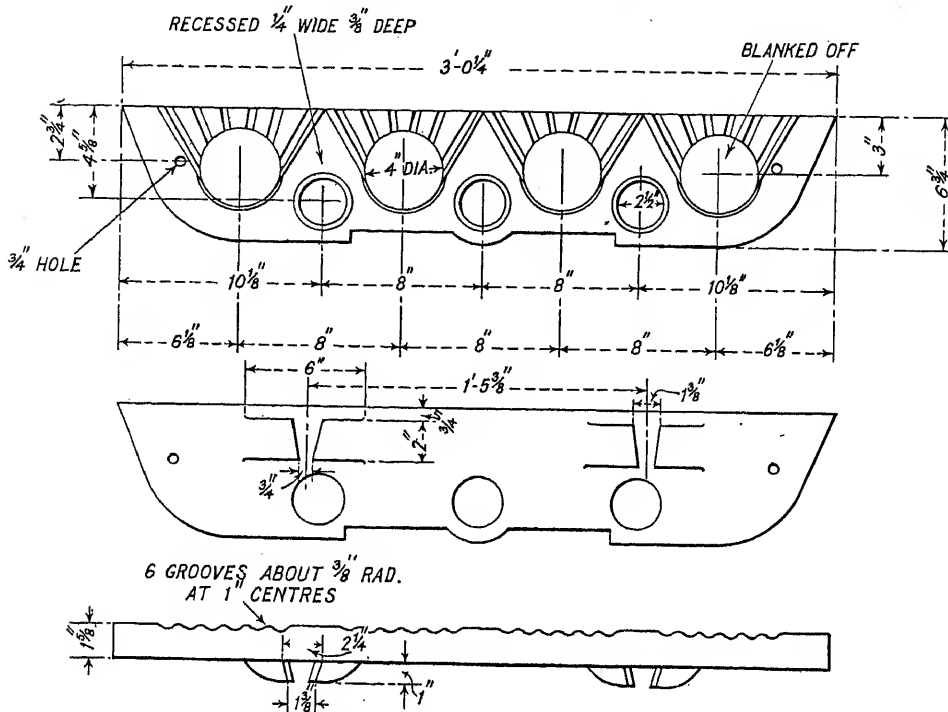


FIG. 7.—CROSTHWAITE PATENT FORCED DRAUGHT FURNACE.

BREEZE COMBUSTION—PRACTICAL CONSIDERATIONS

The suggestion has been made that the best results are obtained by admitting such a quantity of primary air as will lead to the formation of carbon monoxide, which can be subsequently burned to carbon dioxide. The reputed merit of this method of procedure is that it is possible to ensure a high CO_2 content in the waste gases.

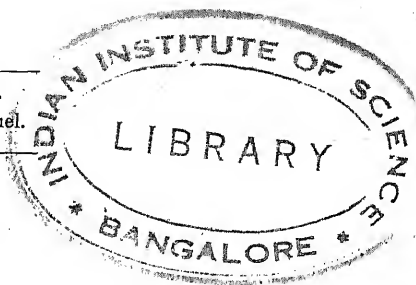
The author's experience of the combustion of coke breeze in forced draught furnaces does not confirm this view. Combustion would be seriously retarded if an attempt were made to restrict the supply of primary air to such proportions as would lead to the formation of carbon monoxide. The resistance offered by the fuel bed demands the introduction of a quantity of primary air sufficient to yield CO_2 .

Analyses of the gases above the fuel bed, when the latter is reasonably clean, and under full blast, show results as high as 16.5 per cent CO_2 and 6.4 per cent CO . With a series of boilers working into a common shaft it is found by continuous recording apparatus that the waste gases can be maintained with an average content of 11 to 12 per cent of CO_2 .

The importance of directing attention to the CO_2 content of the waste gases is strikingly shown in the subjoined table, indicating the lb. of air per lb. of breeze (calorific value 10,080 B.Th.U.), in relation to the percentage of CO_2 .

TABLE 3

Percentage CO ₂ in Chimney Gases.	Percentage Excess Air.	Lb. Excess Air used per lb. of Fuel.
5	250	20.20
6	200	16.10
7	162	13.10
8	133	10.70
9	110	8.86
10	91	7.35
11	75	6.05
12	62	5.00
13	50	4.03
14	40	3.22
15	31	2.50



Basis.—Maximum CO₂, 20 per cent. Air required per lb. fuel, $11.6 \times .695$ lb.

All excess air over that theoretically required carries away heat units which cannot be completely recovered. To work with a theoretical percentage of CO₂ is not desirable, but with higher grade fuel and efficient hand stoking the analyses of the waste gases should be approximately 5 per cent O₂, 13 to 15 per cent CO₂, and CO should be absent. The latter figures are hardly to be expected with breeze of the quality indicated. The essential points arising in the working and control of a battery of boilers where breeze is exclusively used, together with details of the results obtained, will now be given under various headings, for the sake of convenient reference.

MECHANICAL PLANT FOR BREEZE COMBUSTION

There has always been considerable hesitation in resorting to mechanical plant for the combustion of breeze in connection with boiler installations, having regard to the peculiar characteristics of this quality of low-grade fuel.

Recently, however, an installation was put into commission at the Bradford Road Gasworks of the Manchester Corporation. In this installation the breeze arrives by an overhead telfer conveyor, and from this point to the time the ashes and clinker are dumped into carts for removal, the handling is automatically effected.

The plant comprises three Sterling water tube boilers, each having a heating surface of approximately 3400 square feet, and a normal evaporation of 12,500 lb. of water per hour. The working steam pressure is 160 lb. per square inch. The three such boilers are fitted with an economiser of 280 tubes, having a heating surface of 2800 square feet. Each of the boilers is provided with one Underfeed Class A self-contained travelling grate stoker (see fig. 8), with grates 6 ft. 2 in. wide, and 14 ft. $3\frac{3}{4}$ in

11311

661.34

NR4

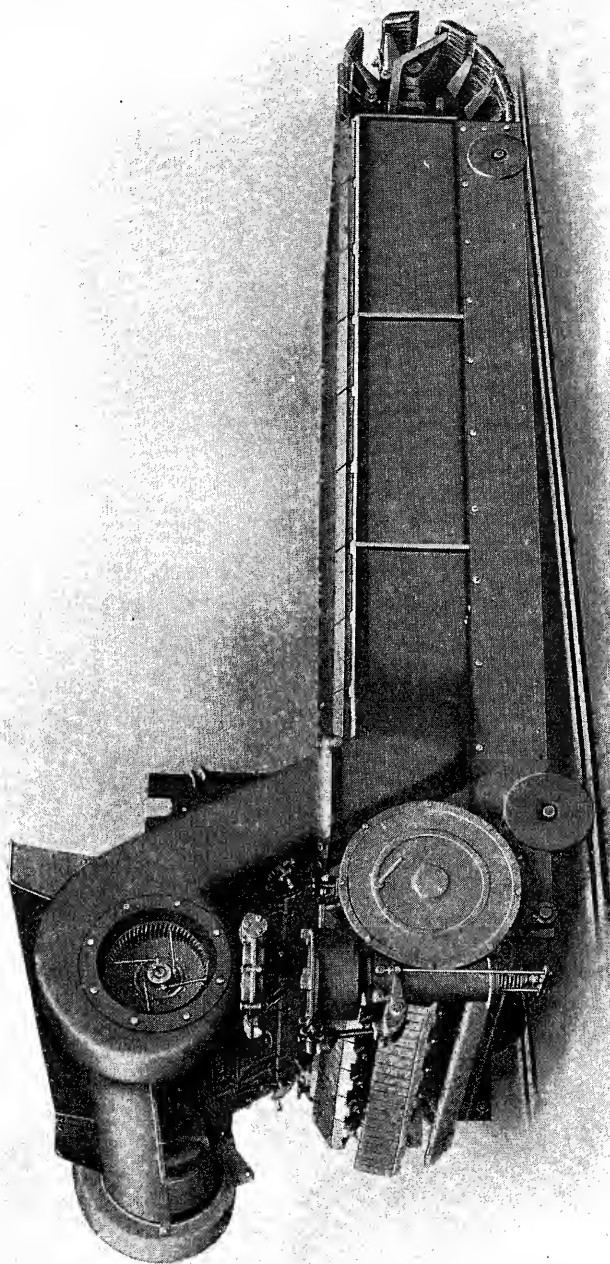


FIG. 8.—UNDERFEED CLASS "A" SELF-CONTAINED GRATE FOR BREEZE.

long, representing 88 square feet of grate area. The power absorbed by the stoker complete, including the fans, is 4 h.p.

In combination with the above mechanical apparatus, is an Underfeed patent water-sluicing arrangement, for handling ashes and clinker as they arise continuously at the end of the stoker. The trough from which the ashes are discharged is built into the boiler setting, the general arrangement of which is shown in fig. 9.

The Underfeed self-contained A-type of impelled draught travelling grate stoker is peculiarly suitable for the combustion of coke breeze. The stoker consists of an endless chain, or grate, and heavy box-shaped fire-bars, mounted on driving chains, connected in the usual way to sprocket wheels, attached to the driving shaft by a special driving mechanism at the front of the stoker. A transverse view of the box-shaped fire-bars shows that they are constituted of heavy framework, having internally a number of small fire-bars. Thus the whole slow moving grate surface consists of a very large number of small fire-bars, with a widely distributed air supply. It is next to impossible for any coke breeze to fall through the grate.

In the event of any of the small bars burning out, they can be replaced at once. The main structure of the grate is not affected. In the process of travelling around the back driven shaft, they open out automatically and drop the ashes into the trough, subsequently coming together on the bottom horizontal travel.

The deposition of the ashes and clinker is a simple matter.

For the forced draught arrangement fans are used, to give the necessary current of air, which is delivered through the trunk to a wind box at each side of the stoker. The total horse power required to drive the fans and supply the air is said to be only 0.3 per cent of the evaporation of the boiler. This is a low figure when contrasted with steam jets, data for which have already been given.

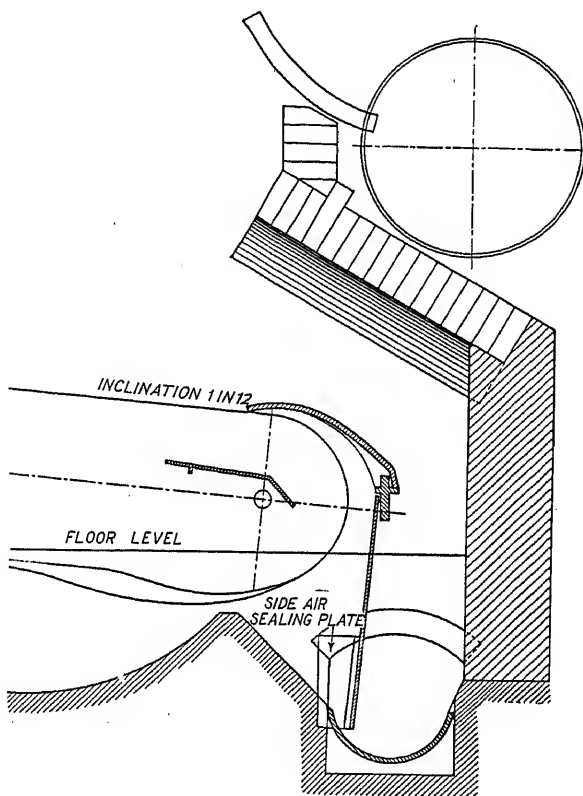


FIG. 9.—ARRANGEMENT OF DISCHARGE OF ASHES FROM TRAVELLING GRATE.

The formation of drift is said to be largely avoided, due to the design of the stoker, and the final arresting of smaller quantities which arise in the sluicing ash conveyors. Approximately 3 tons of breeze per hour are being burned on the installation in question.

Many of the succeeding data, and much of the information which follows, are largely based on the author's practical experience of the operation of a battery of twelve Lancashire boilers (all provided with forced draught furnaces for the combustion of breeze), which are for the most part used for the generation of steam required in the distillation of gas liquor.

BOILER FEED WATER—SOFTENING AND PURIFICATION

This is a large subject, and demands a manual alone—not a succinct treatment such as is here necessitated. The substances occurring in boiler feed water may be broadly classified as follows: (a) those giving hardness; (b) those promoting corrosion; (c) organic matter, and (d) those without action, except when present in high concentrations.

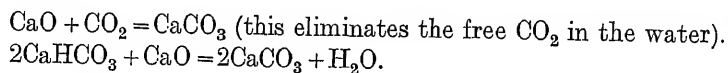
The conducting power of calcium carbonate for heat is one-seventeenth that of iron, and calcium sulphate is one-forty-eighth that of iron. A deposit one-sixteenth of an inch thick, Rankine states, would raise the fuel consumption by 16 per cent; a quarter of an inch thick by 50 per cent, and half an inch thick by 150 per cent. Such deposits are alike dangerous and a source of loss. The efficiency of any type of boiler is roughly proportional to the excess of initial flame temperature over the chimney temperature. The attack of boiler plates may result from over-heating, galvanic action, grooving, pitting, and corrosion. Scale formation leads to over-heating. Galvanic action is caused by an attack between two dissimilar metals, such as iron and copper. This action is very slight in alkaline solution, but traces of acid sensibly accentuate it. To remedy the defect named, it is necessary to bring another metal more susceptible of attack than iron in contact with the boiler shell. Zinc is invariably used. To ensure that there is such contact, an electrical test should be applied. Grooving is caused by bending of the plates, due to alternate heating and cooling. Pitting and corrosion arise from carbonic acid in the presence of dissolved oxygen.

The object to be achieved in the softening and purification of boiler feed water is to convert substances which form a hard scale into those which give a soft scale, and to render all corrosive substances harmless.

IMPURITIES IN BOILER FEED WATER

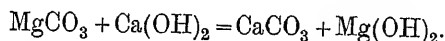
Temporary hardness may be due to calcium and magnesium bicarbonates. Preheating of the feed water is a useful remedy, but it is seldom that a sufficient temperature is reached, or that the preheating is of adequate duration to disengage the whole of the carbon dioxide. Hence calcium carbonate is not completely pre-

cipitated. Generally speaking, it is preferable to have recourse to chemical treatment, which involves the addition of milk of lime. The following reactions represent what takes place :

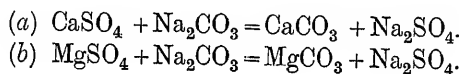


As with calcium carbonate, so to a large extent with magnesium carbonate and bicarbonate. Magnesium carbonate, however, is more soluble in water than the calcium carbonate. In the boiler it is decomposed to magnesium hydrate and carbon dioxide.

Complete dissociation is a function of the temperature attained. Magnesium hydrate in the presence of acids promotes the formation of scale, and not improbably aids corrosion. Magnesium carbonate should be completely removed. In view of its solubility, sufficient milk of lime should be added to give the following reaction :

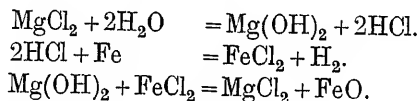


The usual treatment for permanent hardness, which is caused by the chlorides and sulphates of calcium and magnesium, is the addition of sodium carbonate thus :



Calcium sulphate forms a very hard scale, and serves as a binding agent.

Magnesium chloride is usually regarded as one of the most harmful salts occurring in water, decomposing, as it does, in the boiler, and forming free hydrochloric acid. The following equations indicate the reactions :



Two or three methods have been recommended to minimise the effect of these compounds. Some favour the use of tannin ; others consider tannin may cause corrosion of the boiler. Probably the best remedy is to remove magnesium effectively, or to add alkali to the water in sufficient quantity to combine with any acid liberated. Sodium hydrate is best in this case. If sodium carbonate is used, lime must be added to combine with the CO_2 . If lime alone is used calcium chloride and magnesium hydrate result. It has to be remembered that calcium chloride is not altogether harmless. Mere mention can only be made of the necessity for the treatment of water containing oxygen, silica, or iron salts, etc.

Consideration must be given to the cost of treating the water, the debit and credit items being duly taken into account.

There are several methods for softening water available—soda and lime, “permutit,” heat treatment, and caustic soda, etc. The author favours the former on the ground of cost.

WATER-SOFTENING PLANT

The water-softening plant should be simple and automatic as regards construction, ensuring thorough mixing of the reagents with the water. The filtration portion of the plant should be adequate, and efficiently designed.

Fig 10 shows a particularly efficient type of apparatus by which the quantity of reagents is not only measured, but is admixed with a measured quantity of water to be treated. Fig. 11 represents a typical filtration plant for the treated water. In connection with such plant, a definite settlement is provided for, after which the water is subsequently passed through the filtration section, which is constituted of a suitable grid arrangement with wood wool as the filtering medium.

The necessity for chemical control cannot be too strongly urged.

The following statement represents the cost of treating the Metropolitan Water Board's (Kent) water, varying in hardness from 18° to 22°.

COST OF SOFTENING KENT WATER (HARDNESS 18°-22°), BASED ON THE
SOFTENING OF 30 MILLION GALLONS PER YEAR

Cost per 1000 Gallons

	<i>d.</i>
<i>Lime</i> : 1.34 lb. at 37s. 9d. per ton27
<i>Soda ash</i> : 0.74 lb. at £8 per ton63
	<hr/>
	0.90
	<hr/>

CONTROL OF BOILER FEED WATER

Where the steam load is a fairly uniform one, as is the case in most ammonia distillation works, it is desirable to maintain a regular feed of water, as far as the working of the boilers will allow. This does not necessarily mean that the supply to each boiler will be absolutely uniform throughout the twenty-four hours of the day. At certain periods (*e.g.* clinkering), it is usual to reduce the rate of feed slightly until this operation has been completed. Nevertheless, this slight irregularity is not appreciable when viewed from the operation of the installation as a whole. The importance of a regular feed of water to the boilers is enhanced when the arrangements at the ammonia distillation works provide for interchange heating between (say) the waste liquor and the boiler feed water, as is usual at some works.

Trouble from steam "locks" would be prevalent in the event of the flow of boiler feed water to the interchange heaters being checked for too long a period.

STOKING

It is as important to avoid a too deep fuel bed as it is to maintain the full area of the grate uniformly covered with fuel. This is no easy matter with a forced draught furnace where breeze is the fuel. When the fire is dirty, and clinkering time is reached, the bed should be divided longitudinally. Any freshly ignited fuel (and

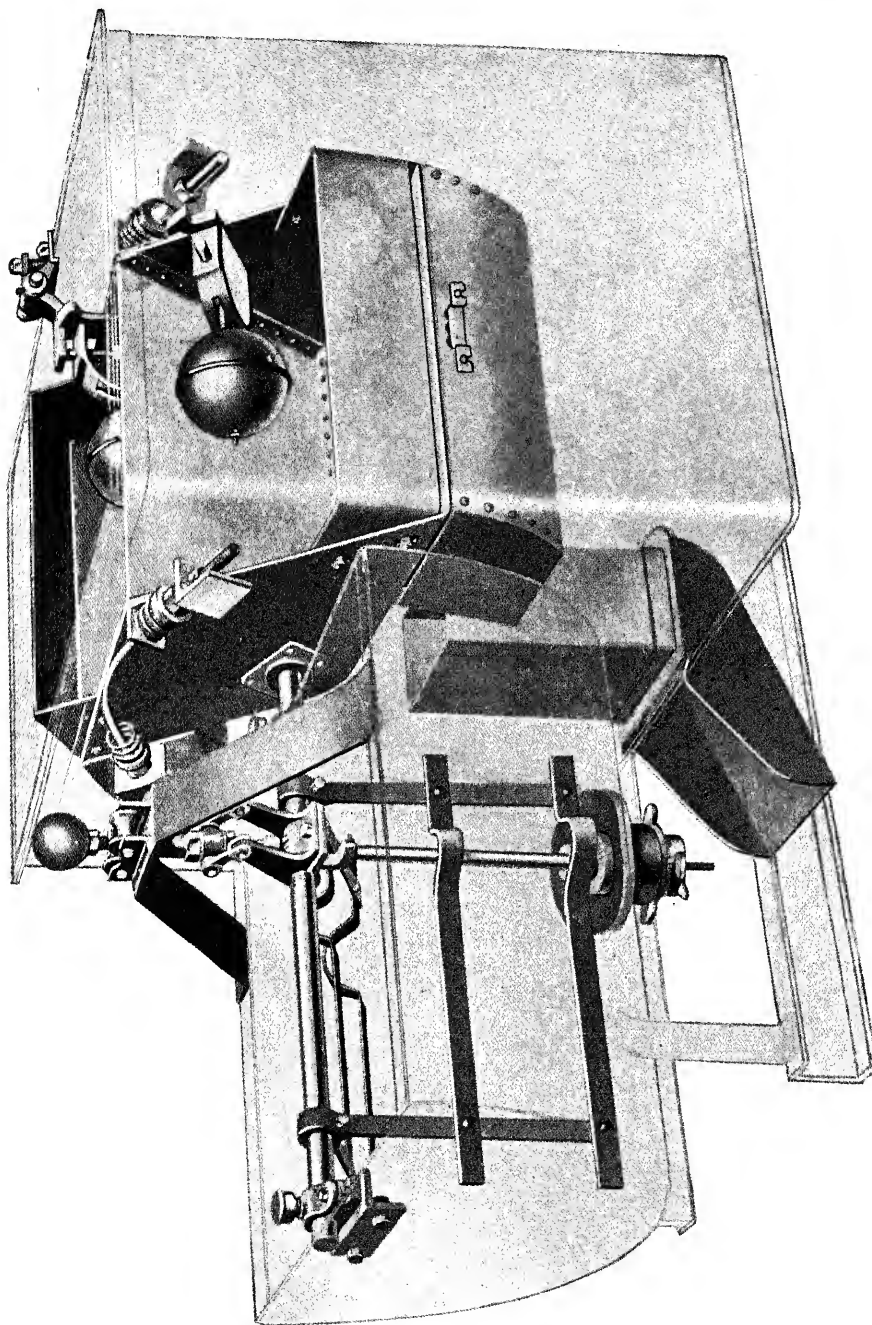


Fig. 10.—WATER-SOFTENING ARRANGEMENT, REAGENT APPARATUS

some should always be present) should be transferred to one side, and the clinker and ashes removed by slicing and drawing from the other side. This done, then the

ignited fuel should be transferred to the clean half of the grate, and the clinker and ashes removed from the remaining dirty side. Subsequently, the ignited fuel should

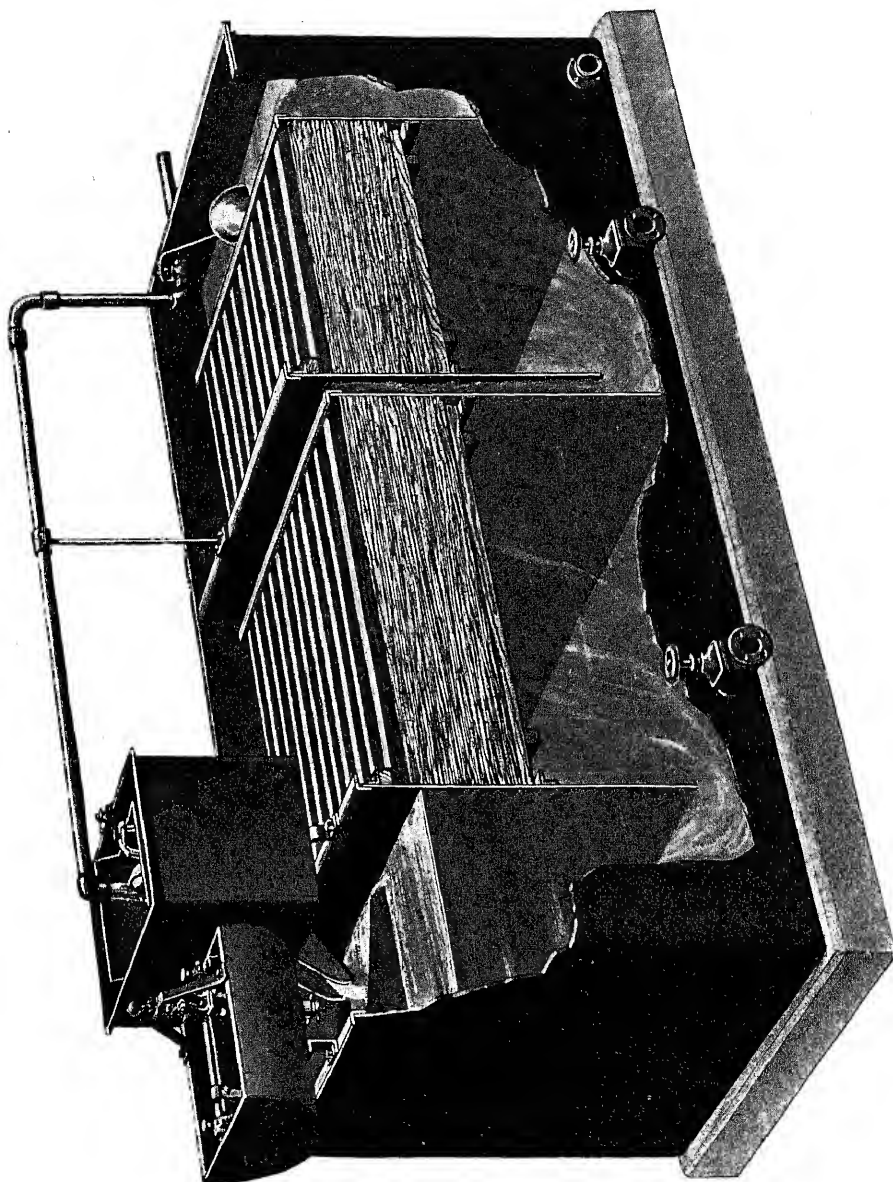


FIG. 11.—WATER-SOFTENING ARRANGEMENT, FILTRATION APPARATUS.

be spread uniformly over the grate area, and a thin layer of breeze evenly distributed throughout. Whether the steam jets should be maintained at a slightly reduced velocity during clinkering, or whether they should be shut off altogether, appears

to be a matter of opinion. Personally, the author favours the passage of steam through the jets at a reduced velocity, as it promotes incandescence of such combustible matter as remains during the clinking operation, and facilitates the ignition of the fresh material after it is charged.

Counterbalanced dampers should be provided, and these should be capable of manipulation from the position where the stoker does the charging. During clinking the dampers should be checked, so as to avoid unnecessary cooling of the tubes and flues, as alike dilution of the flue gases. Meanwhile, the depth of fire should be gradually built up, and the blow-holes through which the induced air is found to penetrate should be covered.

The fire doors have not infrequently to be opened, and thus dilution arises. To fire lightly and often would be sound advice, but for the introduction of redundant air. A compromise of these two diverse influences must therefore be arranged.

The bed of fuel will gradually begin to accumulate in depth, but it is desirable to maintain it at all times uniform, and the grate fully covered. The eventual depth should not exceed six to eight inches.

Firemen who pile up their fires unduly, and increase the velocity of the steam through the blasts, thus promoting all the conditions for producer gas-making, should be taught the folly of such methods. An appreciation of the fundamental principles of steam generation should be made clear, and should be suitably urged.

CLINKERING

Systematic and careful clinking has an important bearing on the efficiency of the results secured. It is usual to clinker the fires twice each eight-hour shift, but owing to the varying quality of the fuel no irrevocable rule can be imposed. With coke breeze high in ash and moisture contents it is necessary to clinker three times each eight-hour shift. When working a series of Lancashire boilers, it is important that the clinking operation should be conducted, as far as the quality of the fuel will allow, to a time table. If ten boilers are working, and the fuel is of reasonable porosity, it is seen that twenty fires have to be cleaned in 240 minutes. This allows 12 minutes per fire. It is usual to clinker and clean the left-hand fires in boilers 1-10 in consecutive order, and then to proceed in the same order with the cleaning of the right-hand fires. The necessity for quick cleaning and recharging is self-evident. It is important that the air tubes should be kept clean. Fine dust passes through the perforations of the bars and must be removed periodically.

ARRANGEMENT OF BOILER FURNACE BRIDGES

Fig. 12 shows that a split bridge is used, the preheated secondary air emerging between the primary and secondary bridges. The secondary bridge assumes the curvature of the tube. The primary bridge rests on a suitable cast-iron plate, supported on the forced draught bars.

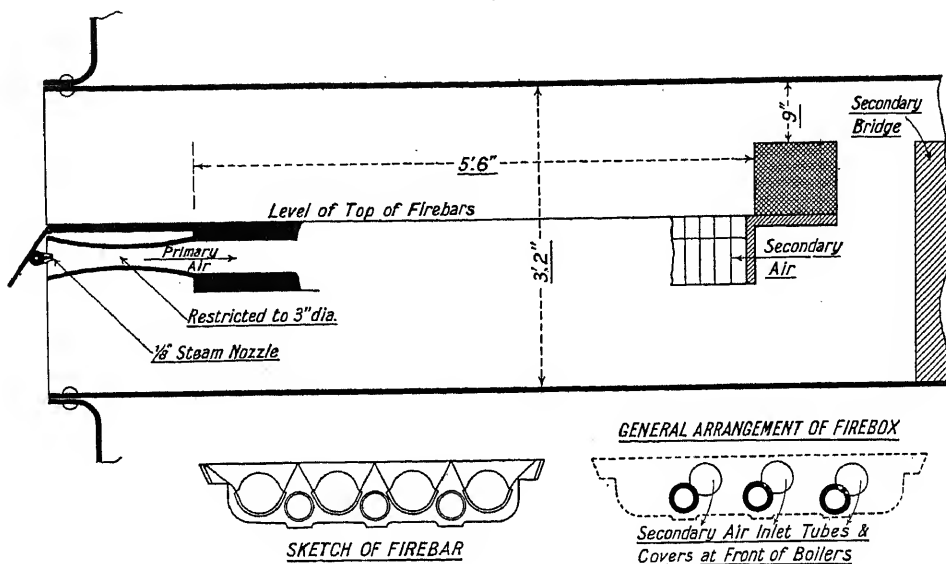


FIG. 12.—GENERAL ARRANGEMENT OF LANCASHIRE BOILER FIRE-GRATE—SPLIT BRIDGE.

FUEL CONSUMED PER SQUARE FOOT OF GRATE AREA

The grate area of each of the boilers is 26 square feet, and the fuel consumed varies between 25 and 30 lb. per square foot per hour. Generally speaking, the author prefers a 5-ft. long grate to a 6-ft. 6-in. one, occasionally recommended by the makers of the forced draught special bars. With a shorter grate it is easier to maintain a uniformly covered fuel bed, and in consequence less dilution of the products of combustion occurs.

EVAPORATION FROM AND AT 100° C.

An average figure is 5.5 lb. per lb. of fuel, dependent on the quality of the breeze. This figure is with boiler plant devoid of economisers, to which aspect reference will be made later.

EVAPORATION CAPACITY OF THE BOILERS

With a battery of Lancashire boilers, each 27 ft. by 8 ft. diameter, the average evaporation from and at 100° C. is 400 to 430 gallons per hour during normal working. As the evaporation of water per lb. of average quality breeze is 5.5 lb., and as the available heating surface per boiler is 725 square feet, a simple calculation (taking the mean evaporation figure) reveals that 1.05 lb. of breeze are burned per hour per square foot of heating surface.

It is said that with hand-fired boilers using coal the combustion of 4 lb. of coal per hour per square foot of heating surface usually gives the maximum efficiency.

It should be remembered in this connection that intensive working of breeze-fired boilers is to be deprecated. The author's experience suggests that there is a distinct relationship between thermal efficiency and productive capacity. A compromise has, however, to be made, and the above rate of working has been found to be satisfactory on the whole.

CONSUMPTION OF STEAM AT THE BLAST JETS

The blast jets have a tendency to enlarge, and periodically it is necessary to

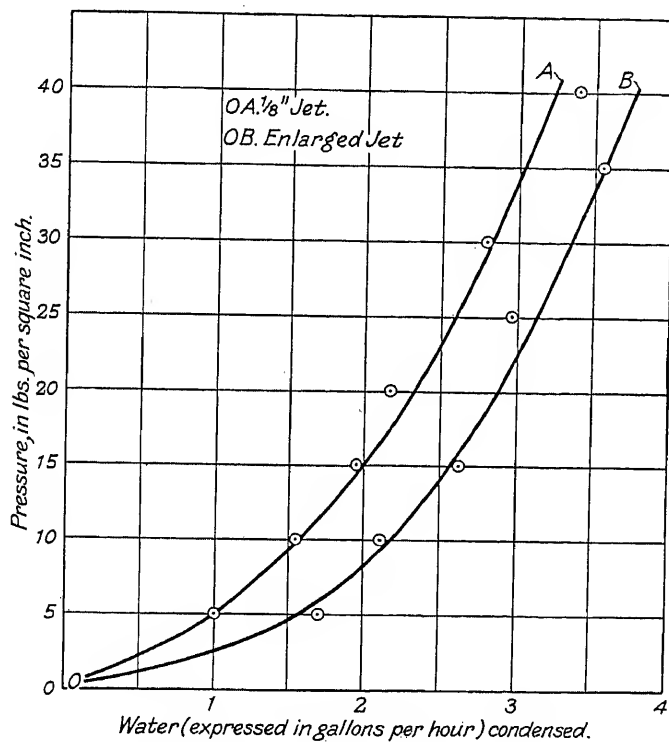


FIG. 13.—GRAPH RELATING TO BOILER JETS.

determine what, if any, enlargement of the orifice has taken place, by testing with a standard gauge. Jets which have enlarged can be bushed, and thus they last for many years, especially if made of suitable gun-metal. The consumption of steam in connection with jets having a $\frac{1}{8}$ -inch diameter orifice is shown in the graph above (fig. 13), as also is the consumption of an enlarged jet.

The steam consumption has been correlated with the consumption of water, and the following table furnishes these data.

TABLE 4
SHOWING CONSUMPTION OF STEAM ON BLASTS IN RELATION TO WATER
EVAPORATED PER HOUR

Pressure at Nozzle. Lb. per sq. in.	Steam used in lb. per hour.		Percentage on Water evaporated.	
	A.	B.	A.	B.
5	80.0	128.0	1.86	2.98
10	121.0	176.0	2.82	4.10
15	159.0	208.0	3.70	4.84
20	184.0	228.0	4.30	5.30
25	208.0	249.0	4.85	5.80
30	225.0	270.0	5.24	6.28
35	242.0	288.0	5.50	6.70
40	264.0	300.0	6.15	7.00

A = $\frac{1}{8}$ -inch jet.

B = enlarged jet. (After twelve months' use.)

Evaporation per boiler per hour, 430 gallons.

Number of jets per boiler, 8.

With good quality breeze it is found that combustion proceeds apace when working with a pressure at the nozzle of 20 lb. per square inch. This represents a consumption of steam of 4.3 lb. per 100 lb. of water evaporated. Based on 46.66d. per ton of steam, it is seen that this represents 11d. for steam per ton of breeze burned. In other words, this implies a cost for steam for the jets of 1.95d. per ton of steam. These figures may usually be increased by 60 per cent if the breeze contains an unusually high percentage of "fines."

DRIFT

The importance of frequent and systematic cleaning of the side and bottom flues of the boilers cannot be too strongly urged, if discharge of grit through the chimney shafts, with its attendant nuisance, is to be avoided. Entrainment of drift (*i.e.* fine dust and grit) by the products of combustion is a direct function of the velocity of the gases. If the gases exceed a speed of six to eight lineal feet per second, it would appear that trouble from drift is inevitable. Obviously, as the flues begin to accumulate dust, so their cross-sectional areas are greatly reduced, with a consequent increase of velocity. If these constrictions of flue areas synchronise with a low CO₂ content in the waste gases, then two potent causes are operating in the creation of the nuisance.

It has been found necessary to clean the side and bottom flues each five weeks.

The removal by screening of all material passing a $\frac{1}{8}$ -inch, or even $\frac{3}{32}$ -inch, screen would certainly assist in mitigating the trouble.

The quantity of flue dust removed at each cleaning represents about four tons, and the ash content varies between 40 and 80 per cent, according to circumstances.

DRAUGHT

One chimney, 4 ft. 6 in. square by 90 ft. high, serves two batteries of three boilers each—that is, six boilers in all.

The suction at the bottom of the shaft is usually $\frac{3}{4}$ -inch water gauge, at the main flue $\frac{5}{8}$ -inch, and the secondary air ports are under $\frac{3}{16}$ -inch suction. The pressure in the primary air conduits is $\frac{1}{2}$ inch, and the products of combustion are in a state of equilibrium immediately above and adjacent to the secondary bridge.

BLOW-DOWN AND PRIMING

The author regards it as essential that all boilers should be “sludged off,” or “blown down” each day. A representative sample of such blow-down water should be taken each day and complete records kept.

Typical results are subjoined :

TABLE 5

Boiler No.	Alkalinity.		Total Solids. Grains per Gall.
	NaOH. Grains per Gall.	Na ₂ CO ₃ . Grains per Gall.	
4	1.12	3.01	144
6	4.69	2.94	289
4	1.12	2.94	263
6	3.36	2.94	413
7	3.36	5.95	495
9	Nil.	2.94	130
11	3.36	5.95	448
1	3.36	5.95	434
2	7.00	4.48	480

It will be appreciated that there are certain salts which are neither scale-forming nor corrosive. The whole of the sodium salts and certain others come under this category. Harmless in small quantities, they promote priming when certain concentrations are reached. M'Gill, who has carried out an investigation on the subject of priming, draws the following conclusions from his experiments :

(1) Sodium salts are mainly responsible for priming. At 10 lb. pressure priming takes place, when the content of Na₂O reaches 105 grains per gallon.

(2) Excess lime will cause priming in the presence of saponifiable fats, which are said to be always present in traces in the boiler.

(3) There is a propensity to priming when solid matter is in suspension.

While the author's practical experience does not confirm the conclusions M'Gill has reached, nevertheless his "findings" are given, as they constitute information of importance.

CLEANING

It has already been pointed out how important it is to clean regularly—say, every five weeks, when working under the conditions which are indicated in this chapter. Usually a battery of breeze-fired boilers works eight to nine months of the year. The period of inactivity is occupied by cleaning at various periods, and at least four weeks are occupied in preparing for the annual inspection, and in carrying out the necessary work which such an inspection entails.

TYPICAL RECORD SHEET, WHICH SHOULD BE PREPARED WEEK BY WEEK

Record sheets of the following character should be kept each week, in order to assist in securing more efficient results. The sources of inefficiency should be clearly indicated.

TABLE 6

BOILER RECORD SHEET

Fuel analysis :

(Weekly Results)

Breeze used	408.0375 tons
Moisture	6.90 per cent
Ash	17.70 "
Calculated calorific value, 11,060 B.Th.U. per lb. of fuel.	
No. of boilers in operation (average)	7.3
Volume of feed water to boilers	612,520 galls.
Less water "blown down"	13,200 "
Volume of water evaporated	599,320 "
Weight of water evaporated per lb. of fuel	6.56 lb.
Temperature of feed water, 50° C.	122.00° F.
Average steam pressure, 83.2 lb.	97.90 lb. abs.
Equivalent evaporation from and at 212° F. per lb. of fuel	7.21 lb.
Thermal efficiency	65.20 per cent
Ash in fuel (natural basis)	17.70 "
True ash in dry clinker	87.54 "
Dry clinker per lb. of fuel	0.202 lb.
Combustible matter in dry clinker	12.46 per cent
Thermal loss due to combustible matter in clinker	3.35 "

CO ₂ in chimney gases (chimney base)	6.70 per cent by vol.
Excess air used	170.00 „
Temperature of chimney gases (<i>t</i>)	211.00° C.
Temperature of boiler house (<i>t</i> ₁)	30.80° C.
Heat carried away in chimney gases	19.00 per cent
Heat converted into dry steam (thermal efficiency)	65.20 „
Thermal loss due to combustible matter in clinker	3.35 „
Heat carried away in chimney gases	19.00 „
Heat unaccounted for (radiation, etc.)	12.45 „

Average volume of water evaporated per boiler per hour 380 galls.

OVER-ALL EFFICIENCY, AND AN EXAMINATION OF THE SOURCES OF LOSS

An over-all efficiency of 62 per cent is possible with a boiler installation having no “economisers,” where breeze is exclusively used. This figure has reference to a year’s working, representing the combustion of several thousands of tons of the low-grade fuel in question.

Itemised general figures are appended :

TABLE 7

Thermal efficiency	62.01 per cent
Loss : (a) Combustible matter in clinker	5.73 „
(b) Heat escaping in flue gases	16.53 „
(c) Unaccounted for	15.73 „
	<hr/>
	100.00 „
	<hr/>

The “unaccounted for” usually consists of the following items :

TABLE 8

	Loss.
(a) Blow-down water (about)	1.00 per cent
(b) Evaporation of water in fuel	5.00 „
(c) Heat of clinker	2.00 „
(d) Heat in flue dust	0.78 „
(e) Combustible matter lost in flue dust (about)	0.95 „
and (f) Radiation loss	6.00 „
	<hr/>
	15.73 „
	<hr/>

The consideration now arises—how can greater efficiency be secured? It will be seen that the principal source of the loss is due to heat carried away by the products of combustion. This represents 16·53 per cent. An installation of economisers would doubtless reduce this loss by at least 8 per cent. The disposition of the economisers would need to be specially arranged. In all probability it would be better to suspend these in a chamber, the bottom section of which was sufficiently capacious to admit of the accumulation of a large quantity of flue dust in such bottom section of the chamber so as to prevent the flue dust from reaching the economiser tubes. The base of the chamber should be of large area, thus affording low velocity of the gases, and allowing of deposition of flue dust. Provision should be made for the removal of all accumulations during full working. An economiser installation designed on some such lines as these, with special supervision given to the scrapers, would conceivably function satisfactorily, and it would appear that the saving to be effected would amply cover the initial capital expenditure on the necessary plant in two or three years' time.

The loss in question can also be reduced by obviating the introduction of unnecessary air through leaky brickwork. All boiler settings using forced draught furnaces should, in the author's view, be securely braced, and the brickwork of the setting should be frequently tarred.

GRADING THE FUEL

The loss of efficiency due to the combustible matter in the clinker and ashes can alone be reduced by securing a better uniformity in the size of the breeze. If breeze is burned containing a percentage of large coke mixed with it, it is certain that a sensible portion of such large coke will be found among the ashes unconsumed. The more regular and uniform the size of the breeze, *caeteris paribus*, the less the combustible matter in the ashes, and *vice versa*.

COST SHEET

Reference to fig. 14, which furnishes comparative costs per ton of steam generated, is interesting. The costs A to H represent published results of several Government factories. In order to render a comparison possible with the results at the ammonia distillation works burning breeze, denoted in the cost sheet by the letters "BW," the cost of coal at the Government plants, instead of being calculated on the original basis of a flat rate of £1 : 0 : 0 per ton, has been computed at the price at which a similar quality coal can be bought, delivered at the ammonia distillation works.

The rate of 13s. 6d. per ton represents the price at which the low-grade fuel—coke breeze—is purchased from the adjoining gas works.

In other respects the figures speak for themselves, and demonstrate the economy resulting from the combustion of coke breeze as contrasted with a higher grade and more costly fuel.

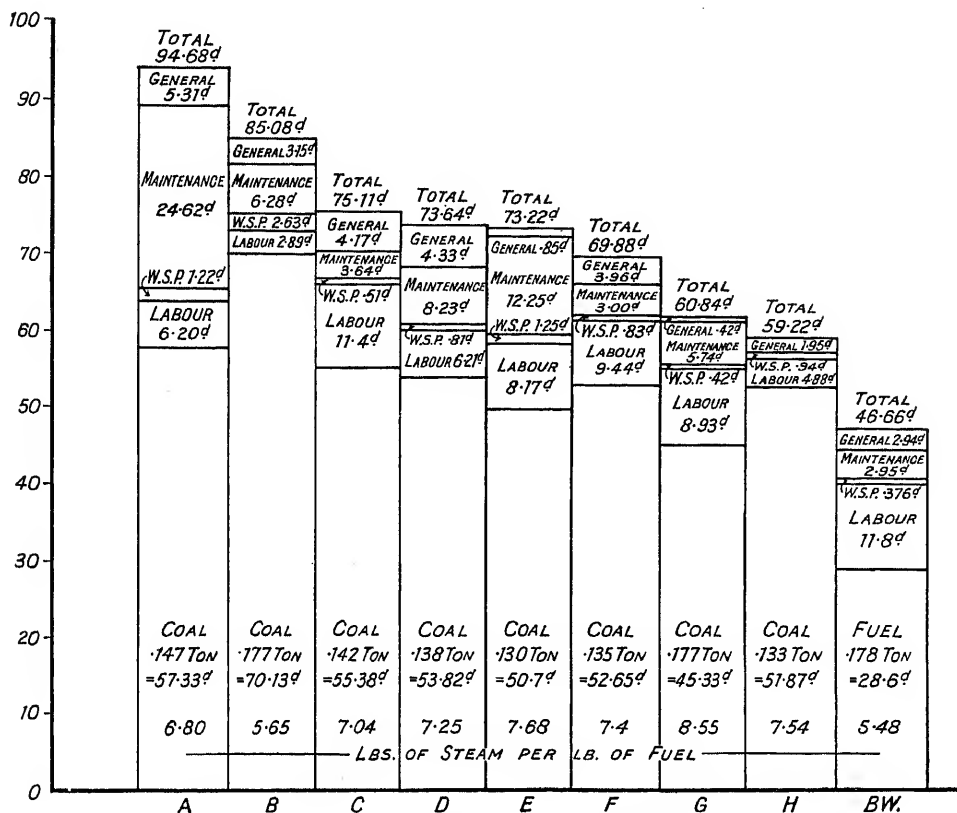


FIG. 14.—COST SHEET—STEAM GENERATION.

GASIFICATION OF BREEZE IN PRESSURE PRODUCERS

Whether the gasification of breeze in pressure producers and the combustion of the resultant producer gas in suitable boilers would be a more efficient and economical method of dealing with breeze is a moot-point.

Trials have been made at one or two works, but the results have not been too encouraging. W. Chaney, in a paper on "Producer Gas and Low-grade Fuel," said: "Gasified alone, the coke dust (all the material passing through a $\frac{3}{8}$ -inch riddle) yielded 95 thousand cubic feet per ton; the calorific value of the gas being 114 B.Th.U. per cubic foot. Unfortunately, the throughput per producer diminished to such an extent as to jeopardise the use of the coke ovens, and the test had to be discontinued."

Prof. Cobb (*Gas World*—Coking Section—April 7, 1917) said: "Gas producers of more orthodox type have been called to aid in the utilisation of low-grade fuel, and more particularly in conjunction with ammonia recovery on the Mond system. But here also there were difficulties. If the fuel was of a non-caking character, such as coke dust, it interposed a considerable resistance, and tended to

blow into channels. Any sudden passage of free air in quantity up one of these channels introduced a special danger in the gas producer, which was absent from the boiler furnace. . . . Moreover, since a deep fuel bed had no power of cohesion it had a liquid tendency to seek its own level, and to run rapidly and suddenly out of the bottom of the lute. This action, by lowering the pressure at the top of the producer, might draw in air through any holes or imperfect joints, with consequent danger."

Rambush, in an excellent paper on "Thermal Losses in the Gas Producer" (Society of Chemical Industry, vol. xl., 1921, 129T), gives the following figures as affecting a coke breeze non-recovery producer :

TABLE 9

Fuel Analysis (Laboratory Examination).	Expected Working Results (Cold Gas Analyses).
Moisture 15%	CO ₂ 7.0%
<i>On dry sample :</i>	CO 24.5%
Ash 30%	CH ₄ 1.1%
Volatile 6%	H ₂ 11.0%
T. carbon 64%	N ₂ 56.4%
Nett B.Th.U. per lb. 9800	Nett B.Th.U. per cubic ft. . . 126.7%
	Cals. per cubic metre . . . 1127.6%
<i>Fuel grading :</i>	Outlet temp. of hot crude gases, as °C. 700
Above ½ inch 15%	Moisture in hot gases, g. per cubic metre (N.T.P.) . . . 90
½ inch to ¾ inch 36%	Percentage carbon in ash . . . 20
Below ¾ inch 50%	Tar yield
Estimation and Calculated Results of Thermal Loss in Percentage of Nett Heat Value of the Fuel.	Sensible Heat Loss in Cals. per Cubic Metre of Gas (N.T.P.).
Moisture loss 2.2%	Percentage CO and CH ₄ . . . 8.1
Tar loss Nil.	Percentage diatomic gases . . 91.9
Dust and soot loss 2.0%	Heat in dry gases 224
Ash loss 10.5%	Heat in steam 30
Radiation loss 1.2%	Total sensible losses, cals. per cubic metre 254
Leakage losses 1.5%	Latent heat of gases 1127.6
Total 17.4%	Total heat of hot gas, cals. per cubic metre 1381.6
Remaining, in hot gas as total sensible and latent heat . . . 82.6%	Ratio = $\frac{\text{latent heat}}{\text{total heat}}$ } . . . 81.6
Sensible heat losses as calculated below 16.3%	Thermal efficiency 62.4

Although it is conceivable that a pressure producer could be designed to overcome the mechanical difficulties which are known to be peculiar to breeze, to which reference has been made, an impartial consideration of the whole question suggests that it is not impolitic to persevere with forced draught boiler installations, adopting economisers for the recovery of the major portion of the available heat in the waste gases, exercising additional care in the matter of reducing combustible matter in the clinker and ashes, and diminishing radiation losses.

Given increased efficiency from the directions indicated, the production of steam from low-grade fuel will be difficult to beat. This is attributable in part to breeze being a relatively cheap fuel.

RECOVERY OF FUEL FROM ASHES

Attention was directed in an article by Dr. H. A. Prager, published in the *Chemical Age*, vol. 8, p. 638, to the recovery of fuel from ashes. Hitherto the known methods of fuel recovery have been the so-called "wet" and "dry" processes. The wet process has been known for several years, but the dry process is a comparatively modern invention. Several types of installations have been constructed for extracting the combustible matter by the washing process, by which advantage is taken of the difference of specific gravity between the coke and the clinker. For certain ashes this method gives good results, but clearly the fuel recovered will be saturated with water, 20 to 25 per cent being not unusual. It would appear that the wet process is unsuitable for ashes containing a large proportion of fine breeze. Further, if the ashes have a porous structure, the difference of specific gravity will not be sufficiently marked to allow of a clean separation, and the fuel obtained will be of indifferent quality.

A method of separation based on an entirely new principle was introduced a few years ago by Ullrich. This is known as the "dry magnetic process." It depends primarily on the fact that practically every coal contains iron as pyrites. During combustion oxides are formed, and these pass completely into the slag. It was found that the clinker containing the iron oxides is attracted in high magnetic fields, whereas the residual combustible matter is not attracted in any way. As the slag need only contain minute quantities of iron, the magnetic separation process is applicable for ashes arising from almost every quality of coal. A quantity of iron in the ashes of about 1.34 per cent, corresponding to a content of about 0.27 per cent in the coal, is adequate. In this connection it should be noted that Fe_3O_4 is a thousand times more magnetic than Fe_2O_3 .

The separation of the fuel from the clinker is effected on the periphery of a rotating drum, inside which are fixed two electro-magnets for the production of the magnetic field. As the ashes pass over the drum, the slag is attracted by the field, while the non-magnetic coke is automatically projected over a partition and discharged into trucks. The underside of the drum is not magnetised, so that the clinker drops off, and is thus automatically discharged. This is illustrated in fig. 15.

The separation is remarkably clean, and a high-quality fuel with a calorific value

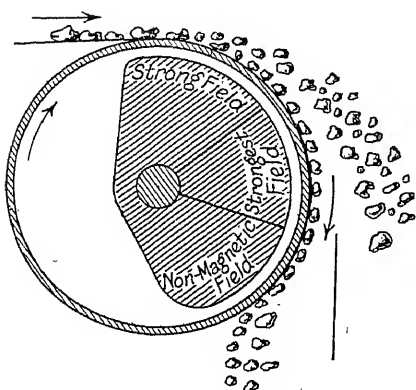


FIG. 15.—MAGNETIC SEPARATOR DRUM.

conical screen, which separates the various sizes, as, for instance, 0 to $\frac{5}{8}$ inch, $\frac{5}{8}$ inch to $1\frac{1}{8}$ inch, and $1\frac{1}{8}$ inch to 3 inch. The recovered fuel is thus obtained already graded.

The ashes pass from the screen to the magnetic separator, and after separation the fuel and clinker are discharged through chutes into trucks. It is known that plants have been erected to deal with 3 tons to 20 tons of ashes per hour (see figs. 16 and 17), one separating drum dealing with about 2 tons of ashes per hour. On an average about 90 per cent of the fuel is recovered.

The power consumption for a small plant is about $1\frac{1}{2}$ h.p. per ton of ashes treated hourly, and about $\frac{3}{4}$ h.p. for the same quantity in a larger plant.

The magnetos are excited by direct current, the consumption being 0.7 to 1 kilowatt per hour per ton of ashes. A small plant can be operated by two men, while three or four men will be required for larger plants.

of 11 to 12 thousand B.Th.U. is secured, irrespective of size, whether as coke or breeze. When it is remembered that breeze constitutes about one-third to one-half of the total fuel content, it will be seen that this is one of the great advantages of the dry magnetic process. Such breeze is largely lost in the wet process.

This process should be erected on the works where the ashes arise, so as to allow of maximum financial advantage. The ashes are charged into a hopper, from which they are fed on to a bucket elevator by means of a shaking chute. The elevator carries the small material to a rotating

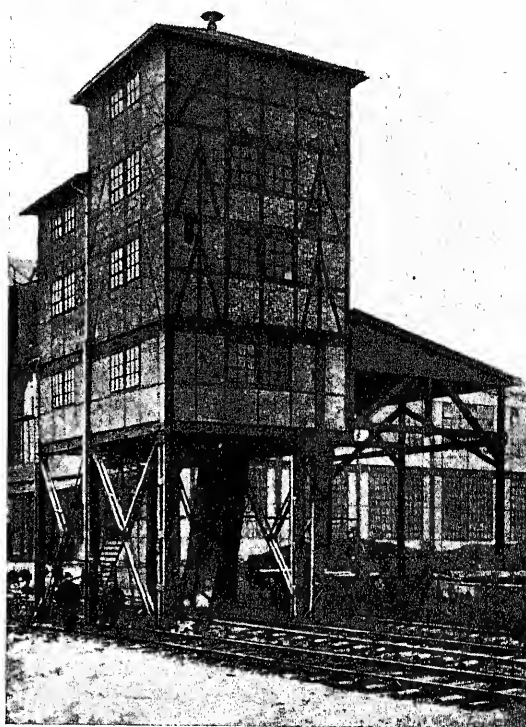


FIG. 16.—FUEL RECOVERY PLANT (3-TON).

FINANCIAL ASPECT OF FUEL RECOVERY FROM ASHES

The financial aspect of the process (and this will be the principal consideration for works which have at present to dispose of large quantities of ashes from boilers) depends essentially on three main factors: (1) character of the ashes to be treated; (2) the extent to which it is possible to utilise the plant throughout the twenty-four hours of the day; and (3) the labour required. When dealing with 48,000 tons of ashes per annum, containing 21.7 per cent of combustible matter, it is computed that the cost of the recovery will be 4s. 6d. per ton.

As has been indicated, given breeze of a reasonably uniform size for combustion in connection with boiler plants, it should be possible to reduce the combustible matter in the ashes to 10 per cent. It appears hardly likely that the treatment of such ashes would constitute a commercial proposition at the moment. It must, however, be borne in mind that the dry magnetic process has many potentialities, and it is not impossible that the operating costs may eventually be reduced. Moreover, the slag which is separated has a monetary value, in that it can be used for making a very satisfactory quality of slag bricks.

Many distillation works not infrequently have large quantities of lime sludge available, and with slag free from fuel at their disposal, it should not be beyond the resources of such works to find some process of utilising these materials in a way which will add to the revenue of the undertaking.

THE RATING OF BOILERS

The capacity of a boiler is invariably stated in "boiler horse-power." A boiler horse-power means the evaporation of 34.5 lb. of water per hour from and at 212° F. To find the boiler horse-power developed, the evaporation from and at 212° F. per hour must be determined, and this figure must be divided by 34.5. Take the figure of 400 gallons of water per hour, mentioned on page 38, as representing the evaporation of water, then:

$$\text{Boiler horse-power} = 4000 \text{ lb.} \div 34.5 = \text{approximately } 115 \text{ h.p.}$$

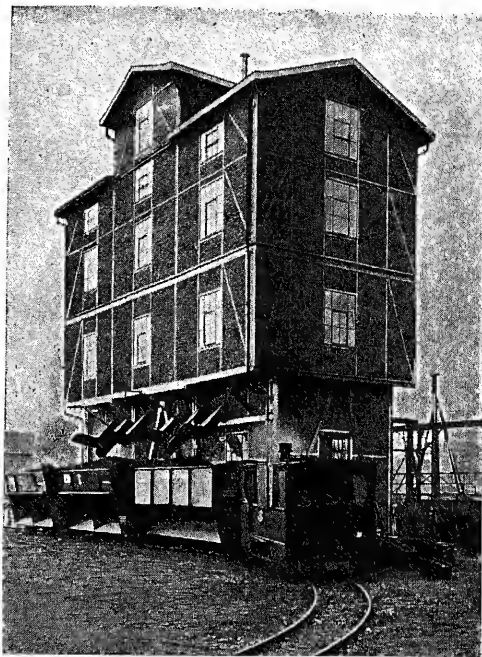


FIG. 17.—FUEL RECOVERY PLANT (12 TON).

The rated horse-power, as stated by boilermakers, is the number of square feet of heating surface afforded by the boiler, divided by a figure of 10, which is assumed to represent the amount of heating surface per horse-power per hour.

In connection with the boilers which have been considered, it will be seen that the heating surface of each boiler is 725 square feet ; hence, the rated capacity is

$$\frac{725}{10} = 72.5 \text{ h.p.}$$

Clearly, the boilers in question are working intensively—indeed, at a capacity far in excess of the usual rating of the boilermaker.

CHAPTER III

THE COMPOSITION OF AMMONIACAL LIQUOR, AND OTHER DATA

THE following analyses give an indication of the average composition of the ammoniacal liquors arising at various works :

TABLE 10

	Gas Works.	Coke Oven.	Blast Furnace.	Shale Works.
	Per cent w/v.	Per cent w/v.	Per cent w/v.	Per cent w/v.
Volatile ammonia . . .	1.4 to 2.5	0.68 to 0.85	0.4	0.9
Fixed ammonia . . .	0.1 „ 0.4	0.3 „ 0.10	0.01	0.3
Total ammonia . . .	1.5 „ 2.9	0.98 „ 0.95	0.41	0.93
(NH ₄) ₂ S . . .	0.8 „ 0.9	0.40	..	0.10
(NH ₄) ₂ CO ₃ . . .	5.0 „ 8.5	1.90	1.1	2.9
NH ₄ Cl . . .	0.5 „ 1.0	0.20	0.006	0.015
NH ₄ CN . . .	0.07 „ 0.3	0.04

The gas liquor as produced in Scotland, generally speaking, contains about 10 per cent of the ammonia as fixed ammonia, as compared with about double the quantity which gas liquors as produced in England contain.

The gas liquor with which the author has had to deal during the last few years has exhibited a remarkably constant CO₂ : H₂S ratio, viz. 7 : 1, while the NH₃ : H₂S ratio has averaged 1.73 per cent : 0.28-0.35 per cent.

The ammonium compounds constituting gas liquor are divided under two heads : (1) Those volatile at ordinary temperatures, viz. ammonium monocarbonate, bicarbonate, sesquicarbonate, hydrosulphide, sulphide, and cyanide. (2) Those fixed at ordinary temperatures, but which can be decomposed by an exchange of alkali base—ammonium sulphate, sulphite, thiosulphate, thiocarbonate, chloride, thiocyanate, ferrocyanide. Gas liquor also contains pyridine bases, phenols, naphthalene, and tarry matter, which vary appreciably according to the extent to which the liquor is separated from the tar in the “wells” at the gas and coke oven works, and whether or not it is filtered to eliminate the floating tar or hydrocarbons.

TAR AND LIQUOR SETTLEMENT AT GAS WORKS

Fig. 18 shows a diagrammatic sketch of tar and liquor connections and is specially arranged to ensure freedom of the gas liquor from heavy and light tarry matter.

The principle of settlement is resorted to.

All delivery pipes are run to the bottom of the wells and are turned tangentially to give a gentle stirring motion to the liquor without splashing.

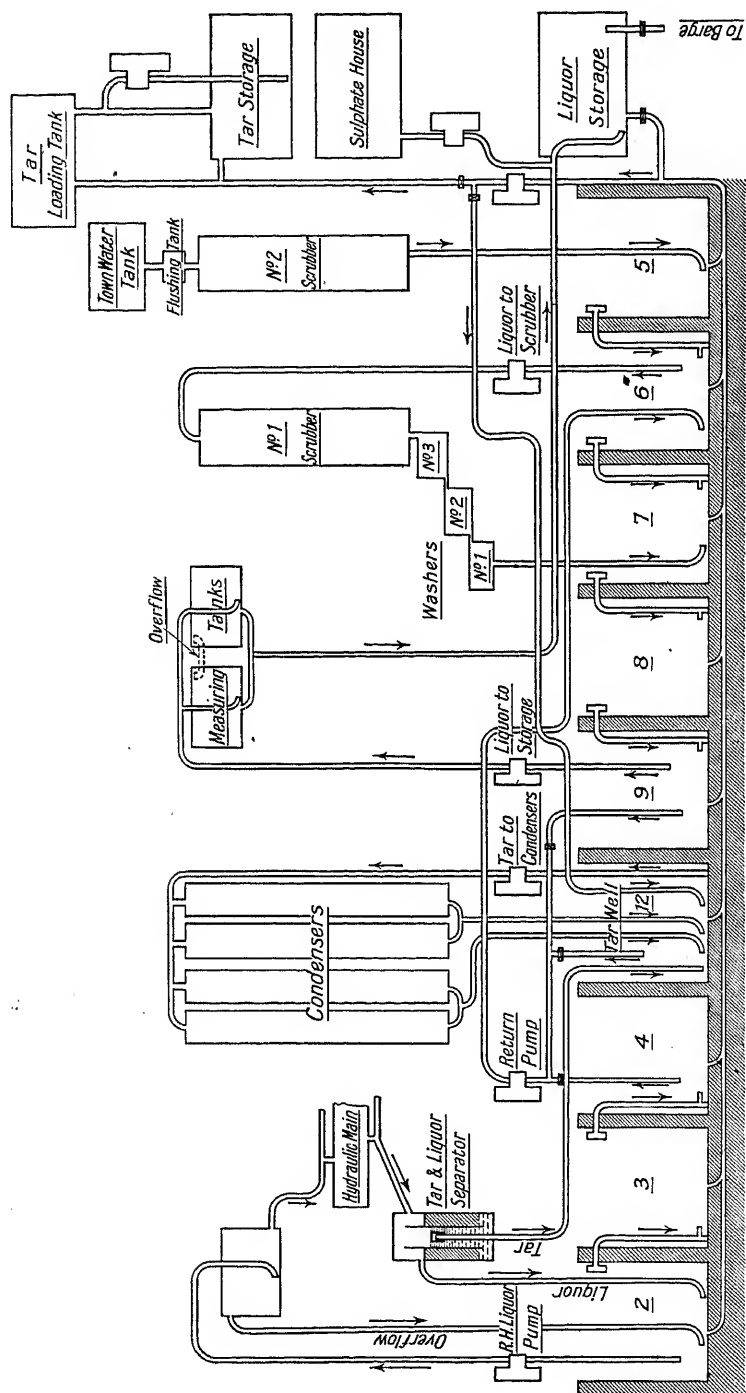


Fig. 18.—SCHEME FOR SEPARATION OF TAR AND LIQUOR.

The tee-pieces at the top of the overflow pipes in the liquor wells intercept light oils in the form of tarry scum. Gas liquor containing tarry matter is disadvantageous in many ways, and too much stress cannot be laid on the necessity for efficient separation of the tar from the liquor and for the adoption of similar methods for the removal of the hydrocarbons specifically lighter than the liquor itself.

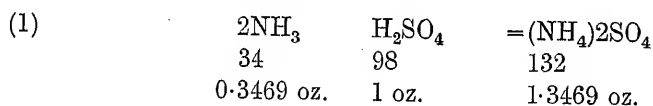
It is not proposed to enter into a lengthy discussion of the methods for the analysis, or evaluation, of gas liquor. This subject has been admirably dealt with in the last few years in the annual reports of the Chief Alkali Inspectors, and the work of Mr. Linder in this connection is well known.

It is recommended that the Chief Alkali Inspectors' reports in question should be consulted for methods of analysis of gas liquor, and also Weyman's *Modern Gas Works Chemistry*.

Many gas works in England still state the content of ammonia in terms of oz. strength, which originally implied the number of ounces of real sulphuric acid required to saturate 1 gallon of the gas liquor. This was the basis of the Wills saturation test, but as it did not contemplate the fixed ammonia, it is now usual to pursue one or other of two methods to arrive at the total content of ammonia.

At some works it is usual to distil off the free and fixed ammonia of the gas liquor into acid which, at 15° C., contains 10 parts by weight of real sulphuric acid in 100 parts by volume, titrating from a burette of 1 oz. capacity graduated in $\frac{1}{16}$ ths of an ounce, and subdivided into tenths (i.e. into $\frac{1}{160}$ ths in all), and to read off the oz. strength direct. In other cases the free and fixed ammonia contents of the gas liquor is distilled into N/1 sulphuric acid, and the ammonia content expressed in terms of percentage of ammonia w/v and a factor used for conversion to oz. strength.

The following data will doubtless be useful as bearing on the oz. strength in its relationship to percentage of ammonia :



(2) 1 oz. strength = 0.3469 oz. of ammonia per gallon.

(3) As 1 gallon contains 160 oz., therefore the parts of ammonia by weight per 100 parts by volume will be

$$160 : 100 :: 0.3469 : x$$

$$x = 0.217.$$

(4) If 0.217 part of ammonia by weight per 100 parts by volume represents 1 oz., then the factor for converting per cent ammonia w/v to oz. strength is

$$1.0 \div 0.217 = 4.61.$$

(5) In London and the South of England liquor is still sold by, or expressed in terms of, butts of 108 gallons.

(6) One degree Twaddell is usually regarded as equal to 2 oz. strength.

- (7) To arrive at the number of butts of liquor (say) of 8 oz. strength required to produce 1 ton of sulphate of ammonia of 25.25 per cent ammonia content.
- (a) One ton of sulphate of ammonia of the above content will contain 0.2525 ton of ammonia, or 9049.6 oz. of ammonia.
- (b) One gallon of 8 oz. liquor contains 2.7752 oz. of ammonia.
- (c) $9049.6 \div 2.7752 = 3260.8$ galls. per ton.
- (d) $3260.8 \div 108 = 30.192$ butts per ton assuming 100 per cent yield.
- (8) To arrive at the number of tons of gas liquor required to produce one ton of ammonium sulphate of given content, divide the percentage of ammonia in the sulphate by the percentage of ammonia in the liquor. Assume 8 oz. liquor (*i.e.* $8 \times 0.2168 = 1.734$ NH_3) and 5.25 per cent NH_3 as the content of the sulphate of ammonia.

$$25.25 \div 1.734 = 14.562 \text{ tons,}$$

or $14.562 \times 224 = 3261.8$ galls.

or $3261.8 \div 108 = 30.192$ butts.

TABLE 11

TABLE SHOWING TONS OF GAS LIQUOR OF VARYING STRENGTHS REQUIRED TO PRODUCE ONE TON OF AMMONIUM SULPHATE, HAVING VARYING STRENGTHS OF AMMONIA

Strength of Gas Liquor.		Tons of Gas Liquor per ton of Ammonium Sulphate based on 100 per cent Conversion Efficiency.							
Per cent. Ammonia.	Oz. Strength.	Ammonia Content of Sulphate of Ammonia.							
		25 per cent.	25.10 per cent.	25.20 per cent.	25.30 per cent.	25.40 per cent.	25.50 per cent.	25.60 per cent.	25.70 per cent.
0.868	4	28.90	29.02	29.14	29.25	29.36	29.47	29.59	29.71
1.085	5	23.03	23.13	23.23	23.31	23.41	23.50	23.59	23.68
1.302	6	19.21	19.29	19.36	19.44	19.51	19.59	19.66	19.74
1.519	7	16.46	16.53	16.59	16.65	16.73	16.79	16.86	16.92
1.730	8	14.45	14.51	14.56	14.62	14.69	14.74	14.80	14.86
1.953	9	12.80	12.85	12.90	12.95	13.00	13.05	13.11	13.16
2.170	10	11.52	11.57	11.61	11.66	11.70	11.75	11.80	11.84
2.389	11	10.46	10.51	10.54	10.59	10.63	10.67	10.71	10.75
2.604	12	9.60	9.64	9.68	9.71	9.75	9.79	9.83	9.87

" BUTTS," AND " OZ. STRENGTH "

It is not improbable that many chemists and technicians will view with apprehension the reference to such units as " butts " and " oz. strength." The author does not desire to perpetuate unduly such units.

Unfortunately they are still live terms in the South. It is hoped, however, that a drastic endeavour will be made before long to dispense with them.

COMPOSITION OF AMMONIACAL LIQUOR, AND OTHER DATA 55

These standards have doubtless served a useful purpose in the past, but it would appear that they are uncalled for in these days.

CHARACTERISTICS OF AMMONIACAL LIQUORS

The composition of ammoniacal liquor depends to a large extent on the impurities in the coal to be carbonised or coked ; on the temperature of carbonisation ; on the extent to which the liquor has been exposed to the air (which causes oxidation of the sulphides to thiosulphates) ; on the temperature to which the liquor has been subjected, and on the extent to which chlorides are present in the coal.

The proportion of fixed ammonia in the liquor decreases with the process of condensation. It is highest in the hydraulic main, and lowest at the end of the washing process. In the hydraulic main the fixed ammonia content is approximately 50-60 per cent ; in the condensers about 5 per cent, and in the washers about 4 per cent.

The under-mentioned table reveals the properties and composition of various samples of liquor from different coals and from different points in the condensing and scrubbing plant.

TABLE 12

TABLE SHOWING THE PROPERTIES AND COMPOSITION OF VARIOUS SAMPLES OF LIQUOR FROM DIFFERENT COALS, AND FROM DIFFERENT POINTS IN THE CONDENSING AND SCRUBBING PLANT. (From Alkali Inspector's Annual Report)

	Condensed Liquor, passed through Scrubber.	Another Sample taken at different Time.	Same Liquor, exposed to Air, High Temperature.	Hydraulic Main Liquor.	Part Condensed Liquor, part Hydraulic Main.	From "Standard" Washer before Purifiers.
Colour	Yellow	Yellow	Bright orange	Dull yellow	Yellow	Colourless
Spec. grav. = deg. Tw. at 15°·5	4·5	4	3·5	2	3	4·5
Ounces by distillation test .	10·00	8·25	6·90	5·60	6·60	10·00
Ounces by saturation test .	8·30	7·00	4·25	3·40	4·90	10·00
Am. sulphide, grains per gallon	960	1216	630	576	220	1180
=NH ₃ "	480	608	315	288	110	590
Am. carbonate "	2030	1156	800	..	1692	2540
=NH ₃ "	720	410	283·5	193	600	900
Am. thiosulphate "	195	182	236	..	174	Trace
=NH ₃ "	45	42	52·5	35	40	..
Am. sulphate "	..	Trace	39	..	9	..
=NH ₃ "	10·5	6	2·5	..
Am. sulphocyanide "	338	243	472	..	90	..
=NH ₃ "	75	54	105	36	20	..
Am. ehloride "	567	403	891	..	693	32
=NH ₃ "	180	128	283·5	301	220	10·1
Am. ferroeyanide "	Trace	31	..
=NH ₃ "	7·5	..
Total ammonia	1500	1250	1050	860	1000	1500
Percentage of fixed ammonia .	20	18·3	43	44	29	0·7
Ammonia expressed as ewt. sul- phate per 1000 gallons . .	7·5	6·00	5·00	4·10	4·85	7·30

TABLE 13
TABLE SHOWING THE PROPERTIES AND COMPOSITION OF VARIOUS SAMPLES OF GAS LIQUOR OBTAINED FROM THE SAME
COAL, BUT TAKEN FROM DIFFERENT POINTS IN THE CONDENSING AND SCRUBBING PLANT

Colour	Condensed in the Hydraulic Main.	Another point of the Hydraulic Main.	Liquor from first Condenser Column.	From second Condenser Column.	From third Condenser Column.	From fourth Condenser Column.	From first Washer.	From finishing Washer.
	Very dull orange, turned black in air	Ditto	Colourless	Almost colourless	Brown-red from light tar-oils	Dark brown, from tar-oils	Colourless	Colourless
Specific gravity in deg. Twaddell at 15° C.	2½	2½	7	15	23	24	4½	2
Ounces by distillation test	6.1	6.0	16.2	36.1	53.0	58.0	16.5	8.3
Ounces by saturation test	2.7	2.8	15.9	35.7	52.5	57.4	16.1	8.1
Am. sulphide, grains per gall.	364	440	2430	5000	7905	8,440	1592	1220
= NH ₃	182	220	1215	2500	{ NH ₃ }	4,220	796	610
Am. carbonate	564	510	3384	8120		12,126	4512	1690
= NH ₃	200	81	1200	2880		4,300	1600	600
Am. thiosulphate	122	82	Trace	125	352	765	230	135
= NH ₃	28	19	Trace	41	81	176	51	31
Am. sulphate	8	34
= NH ₃	2	9
Am. sulphocyanide	112	130	91	Trace	112	27
= NH ₃	25	29	2	Trace	25	6
Am. chloride	1552	1455	119	155	201	107	88	38
= NH ₃	493	462	38	50.	64	34	28	12
Am. ferrocyanide	Trace	22	41	125	375.
= NH ₃	5	10	30	90
Total ammonia, grains per gall.	930	920	2460	5480	8080	8,820	2500	1260
Percentage of fixed ammonia in the total ammonia	59 p.e.	56 p.e.	1.8 p.e.	1.85 p.e.	2.2 p.e.	3.4 p.e.	4.2 p.e.	4.0 p.e.
Ammonia expressed as cwt. of sulphate per 1000 gallons	4.5	4.5	11.9	26.6	39.3	42.8	11.8	6.1

COMPOSITION OF AMMONIACAL LIQUOR, AND OTHER DATA 57

The table on preceding page, taken from the Alkali Inspector's Annual Report, is interesting as showing the properties and composition of various samples of gas liquor obtained from the same coal, but drawn from different points in the condensing and scrubbing plant.

As regards the oxidising influence of the air on gas liquor, to which reference has already been made, the following analyses by Linder are illuminating in this connection :

TABLE 14

In One Litre there was contained	Immediately after obtaining the Liquor.	After Six Months' Storage.
	Grams.	Grams.
Ammonia total	27.52	29.80
As easily decomposable salts	21.76	19.20
As not easily decomposable salts	5.76	10.60
Sulphur total	8.55	9.12
As sulphate	0.70	4.50
As sulphocyanide	23.40	57.90
As thiosulphate	3.10	16.40
Carbon dioxide	25.51	21.22
Hydrogen sulphide	6.61	2.05
Hydrogen cyanide	0.68	0.00
Ferrocyanide calculated as hydrogen cyanide	0.068	0.00

Having regard to the fact that extra expense is incurred in decomposing the fixed ammonia salts (see page 277), the necessity for treating the liquor as early as possible after it has been produced is obvious.

In the 43rd Annual Report of the Chief Alkali Inspector further reference is made to the constitution of ammoniacal liquors, particularly as relating to the amount and distribution of cyanogen compounds, both in coke oven liquors and their distillation products.

Coke oven liquors differ materially from those produced and treated in gas works residual plants.

Excess of air (as has been indicated already) promotes oxidation of ammonium sulphide to ammonium polysulphide, which parts readily with its sulphur to ammonium cyanide (which is one of the simple products of condensation, as an examination of various liquors will reveal), and thus thiocyanate is formed.

The secondary oxidation reactions are dependent on the length of time during which the liquor is stored. As the storage accommodation at coke ovens is usually restricted, so the secondary reactions are limited in consequence, and hence coke oven liquors have a high content of cyanogen volatile as hydrocyanic acid. It has been shown by Linder that the liquors proceeding from the condensation of the steam and fuel gases from the sulphate of ammonia saturators after extraction of the

ammonia, owing to the high proportionate solubility of hydrocyanic acid, contain this substance when drawn from the cold end of the condensing apparatus in a very marked amount; indeed, so great as to need special precautions in handling. In one case cold devil liquor was found to contain 0·7 per cent to 1 per cent of this poisonous compound. Tables 15 and 16 are interesting as bearing upon the foregoing subject.

TABLE 15
COKE OVEN LIQUORS
(As fed to Still)

Type of Oven.	In Liquor. Grams per 100 c.c.				Saturator Gases. Per 100 Vols. (By theory.)		
	Total Ammonia.	Carbonic Acid.	Sulphuretted Hydrogen.	Hydrocyanic Acid.	CO ₂ .	H ₂ S.	HCy.
<i>Hüssner</i> :							
1 sample	·935	·946	·292	·058	66·6	26·7	6·7
<i>Koppers</i> :							
Average of 3 samples .	1·261	1·246	·173	·018	83·1	14·9	2·0
<i>Otto-Hilgenstock</i> :							
Average of 12 samples .	·841	·892	·230	·041	71·0	23·7	5·3
<i>Semet-Solvay</i> :							
Average of 7 samples .	1·202	1·111	·193	·035	78·4	17·6	4·0
<i>Simon-Carves</i> :							
Average of 7 samples .	1·018	1·206	·132	·027	84·8	12·1	3·1
Average of 30 samples	1·012	1·053	·195	·035	77·3	18·5	4·2

GAS LIQUOR STORAGE TANKS

As to the form of the tanks and the general arrangement for the storage of gas liquor, practice varies considerably throughout the country. The shells of Lancashire boilers are used in many cases for the storage of gas liquor, and it is known that they have served fairly well for this purpose.

It is usual at small and medium-size works to elevate the boilers on pier walls, or even on the walls of the still-house itself, so that the liquor to be fed to the still can gravitate to a constant feed tank provided with suitable float or special ball valve for ensuring a uniform feed of liquor to the stills. Experience suggests, however, that sectional plated cast-iron tanks are preferable, and are calculated to have a much longer life than second-hand steel boiler shells.

A typical cast-iron tank for a works distilling tolerably large quantities of gas liquor per day is shown in fig. 19. Two such tanks are usually allocated to one unit. One contains the gas liquor for direct supply to the stills; the other is available for filling in the interim. Thus the tanks can be used alternately, and generally are of such a capacity as to afford twelve hours' supply. Each tank is approximately

TABLE 16
CYANOGEN COMPOUNDS IN COKE OVEN LIQUORS
(As fed to Still)

Liquor.	Grams per 100 c.c.						Per cent Total Cyanogen to Ammonia.
	Ammonia.		Cyanogen in Terms of HCy.				
	Total.	Cyanide.	Thiocyanate.	Ferrocyanide.	Total.	Cyanide.	
Maximum cyanogen	1.109	.048	.062*	Nil	.110	43.5	9.0
Minimum cyanogen	1.470	.011	.018	Nil	.029	38.0	2.0
Average (24 liquors)	1.014	.034	.021	Nil	.055	62.0	5.4

* Thiocyanate high; this is attributed to the fact that the sulphate plant was idle 2 days before sample was taken; liquor, therefore, would be partially oxidised.

COKE OVEN WORKS

Devil Liquors

Devil Liquor.	Average Temperature F.	In Devil Liquor. Grams per 100 c.c.			Per 100 Volumes of Gases condensed (calculated).			Saturator Gases HCy per cent. (By theory.)
		Carbonic Acid.	Sulphuretted Hydrogen.	Hydrocyanic Acid.	CO ₂ .	H ₂ S.	HCy.	
COLD:								
1. One sample. 50° F.	50	3.720	.516	.733	67	12	21	4.6
HOT:								
2. Average of 3 samples. 90° F. to 100° F.	93	.064	.017	.081	29	10	61	4.9
3. " 9 samples. 140° F. to 170° F.	157	.065	.011	.023	56	12	32	3.7
4. " 5 samples. 180° F. to 200° F.	188	.024	.002	.012	53	5	42	5.3

25 ft. \times 15 ft. \times 15 ft. The side plates are flanged, and are 5 ft. square by $\frac{3}{4}$ in. thick, and are arranged with centrally placed circular stiffening ribs from which diagonal stiffening arms radiate. The flanges are $\frac{7}{8}$ in. thick, and are provided with stiffening webs. All flanges are machine finished. The joints are smeared with red and white lead, embedded in which are lengths of twine. Two tie-rods $1\frac{1}{2}$ in. in diameter pass through each of the first two tiers of side plates, suitable bosses being cast on the horizontal centre line to receive these tie-bolts. The top tier of plates are braced by a single $1\frac{1}{2}$ -in. diameter tie-bolt placed in the centre of the plate. The bottom plates are of the same size as the side plates, but are provided with a stiffening web in the centre, running parallel with the flanges. Thus the flanges and stiffening ribs are arranged at about 2 ft. 6 in. centres. The bottom of the cast-iron storage tank is supported by R.S.J.'s placed under the flanges and stiffening ribs at the centres just named. To prevent sagging of the tie-rods, vertical angles are provided.

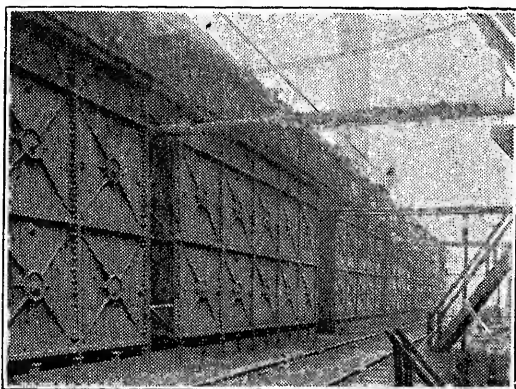


FIG. 19.—GAS LIQUOR TANKS.

The outlet from the tanks is 6 in. in diameter placed 18 in. from the bottom of the tank. At the bottom of the tank a suitable pipe is fixed which is led to a separator for effecting the separation of any tar which has settled out from the gas liquor. A device is also fitted for running off light tar, and hydrocarbons, etc. (see fig. 20).

The tanks are provided at the top with deal joists and are covered with deal boards. From the top of the tank a 6-in. diameter vent pipe leads to a small tower scrubber, which is packed with suitable filling, and provided with an automatic water feed so that the vented ammoniacal air can be thoroughly washed, and the ammonia arrested. The delivery pipe from the gas works liquor wells is led to within a few inches of the bottom of the tanks, so as to reduce the agitation of the gas liquor to a minimum.

The connections from the storage tanks to the gas liquor pumps are made in cast-iron pipe of the turned and bored type. The machining of these pipes is done to a gauge, and they are remarkable for their freedom from leakages.

The tanks are provided with floats which are connected by wire cord over suitable pulleys with a gauge board placed in the still-house, by which the rate at which the liquor is being pumped to the stills can be accurately gauged and determined. This constitutes an additional check on the liquor measuring device fixed adjacent to the stills.

The use of a non-volatile oil on the surface of the gas liquor is a means by which loss of ammonia can be prevented.

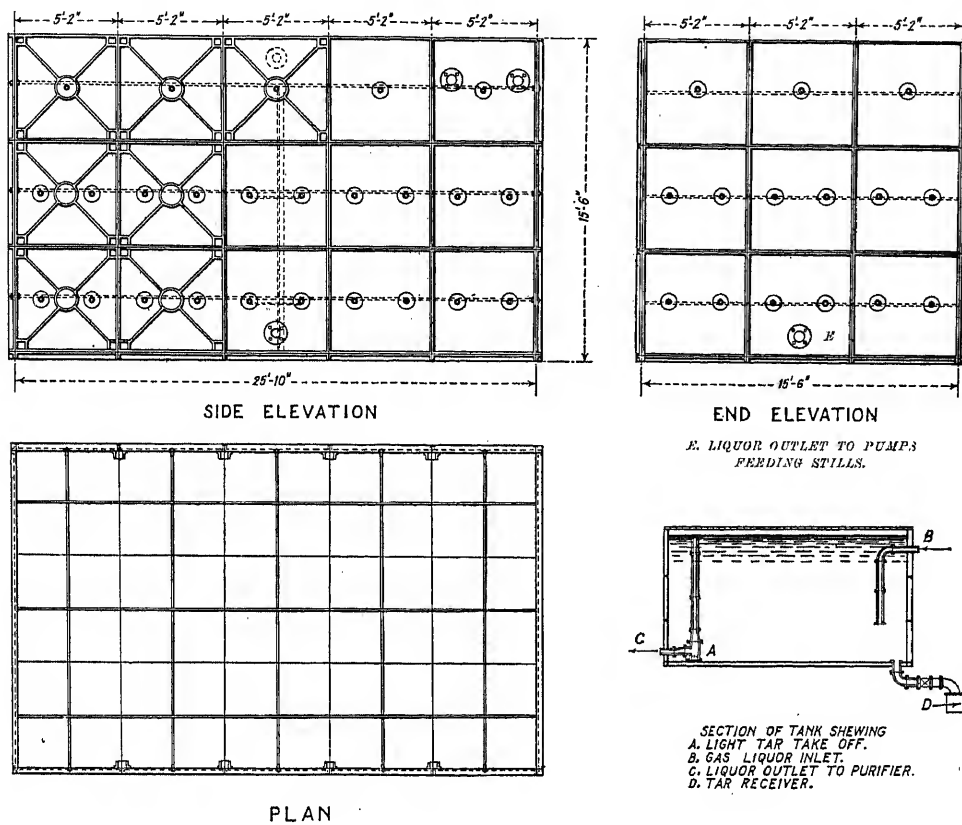


FIG. 20.—CAST-IRON GAS LIQUOR TANKS, SHOWING DEVICE FOR REMOVAL OF "LIGHT" AND "HEAVY" TAR.

CHAPTER IV

A GENERAL DISCUSSION OF PROBLEMS AFFECTING DISTILLATION AND DISSOCIATION

WHETHER the disengagement of ammonia from ammoniacal liquors is essentially a distillation operation, or whether it can be more correctly defined as dissociation, is a matter on which a definite opinion does not appear to have been expressed.

A polemical discussion on the subject by some of the authorities in physical chemistry would be appreciated.

Distillation is usually regarded as the process by which a separation is effected of a volatile liquid from a non-volatile substance, or what is more usual, a separation of two or more liquids of different volatility.

The principles involved in fractional distillation are fundamentally the elementary laws of physics and thermodynamics. It may be urged that, broadly speaking, the same principles apply in the distillation of ammoniacal liquors. This is true, but many of the so-called physical laws governing fractional distillation have a very limited application to dissociation. Solutions of ammonia of such strengths as are typical of normal gas liquor behave generally according to Henry's law. This law provides that the vapour pressure (P) of a volatile solvent and solute is expressed by $P = ax$, in which a is a constant and x the molecular fraction of the solute in relation to the solvent. In the case of ammonia solutions a for accurate values has to be experimentally determined by ascertaining P either by chemical analysis of the vapour over a solution, or by the static method of determining vapour pressure.

For ammonia and water the values of a are well known, ammonia being of immense industrial importance in the manufacture of soda ash by the ammonia-soda process. The values show, however, a slight deviation from those as calculated by Henry's law, owing to the ammonia chemically combining with the solvent to form an easily dissociated ammonium hydrate.

In addition, the deviation of experimental and calculated values increases with decreases of concentration. Such deductions as may be made from the above law apply, of course, only to conditions in a certain section of the gas liquor still, probably the lower portion of the free still, where, owing to the previous decarbonation and desulphurisation, the liquor assumes that of an ordinary ammonium hydrate solution. Although the gas laws allow of a ready interpretation of conditions obtaining in the still, and indeed assist in affording certain fundamentals of design, a point is reached at which further progress in this direction can only result from patient observation of details such as can alone be obtained from practical working conditions.

THE RELATION BETWEEN THE COMPOSITION OF VAPOUR AND LIQUOR IN DISTILLATION

Emil Piron (*Chem. and Met. Engineering*, vol. 26, February 15, 1922), in an article on the subject of "The Relation between the Composition of Vapour and

Liquor in Distillation," reviewed an important physical consideration relating to ammonia distillation. He said :

" If s is the proportion of one component in a mixture such as a gas liquor, and S the proportion of the same component in the vapour distilled from this liquor, the relation between the composition of the vapour and liquor in regard to the considered component is

$$k = \frac{S}{s}.$$

In industrial distillation, liquors are either concentrated or exhausted, and in both cases the strength of the liquor passes through a number of stages of different concentration. Knowing the values of k corresponding to these different stages, we are in a better position to study the design of the distillation apparatus and to control its operation.

DETERMINING DISTILLATION CONSTANT EXPERIMENTALLY

One way in which the values of k can be obtained is to submit a relatively large quantity of liquor to distillation, taking very small fractions of the distillate, so that the composition of the liquor can be considered as constant during the distillation of each fraction. An analysis of the liquor and of the corresponding distillate will give corresponding values of s , S , and k .

In Table 17 a distillation test on 1000 c.c. of ammonia liquor, as obtained by coal distillation, is given as an example. It will be noted that the value of k varies with the strength of the liquor.

OBTAINING THIS VALUE BY CALCULATION

On the other hand, in distillation problems we need an ideal or average constant of k , which, when applied to the problem, will give a result conforming to the actual. This ideal value of k can be found by the following method. Suppose we distil out a quantity Q of liquor, containing a proportion s of a component, an infinitesimal quantity dQ . The proportion of the same component in the distillate at the beginning of the distillation is S , at the end $S - dS$. The average strength of the distillate is $S - \frac{1}{2}dS$, while the quantity of the component we have in the distillate is $(S - \frac{1}{2}dS)dQ$.

During the distillation the proportion of the component in the liquor changed from s to $(s - ds)$, the quantity remaining in the liquor after distillation being $(Q - dQ)(s - ds)$.

Hence the relation

$$Qs - (Q - dQ)(s - ds) = (S - \frac{1}{2}dS)dQ,$$

which gives, after neglecting the differentials of the second order,

$$\frac{dQ}{Q} = \frac{ds}{S - s},$$

or, if the initial quantity of liquor was Q_0 ,

$$\log_e Q_0 - \log_e Q = \int_{s_0}^s \frac{ds}{S-s}.$$

If $\frac{S}{s} = k$ (constant), then

$$\log_e Q_0 - \log_e Q = \frac{1}{k-1} (\log_e s_0 - \log_e s)$$

or

$$\left(\frac{Q_0}{Q}\right)^{k-1} = \frac{s_0}{s}, \quad (1)$$

which gives the desired value of k .

TABLE 17
DISTILLATION TEST ON 1000 C.C. OF AMMONIA LIQUOR

Liquor in Distillation.				Distillate.			
Quantity, c.c.	Volatile Ammonia.			Quantity, c.c.	Ammonia.		$k = \frac{S}{s}$
	g. Total.	g. per Litre.	Average.		g. Total.	g. per Litre.	
1000	7.7200	7.7200					
975	5.9540	6.1060	5.835	25	1.7660		
970	5.4960	5.5630		5	0.4580	91.590	15.7
900	1.7850	1.9830		70	3.7110		
895	1.6462	1.8470	1.915	5	0.1388	27.762	14.5
800	0.4735	0.5920		95	1.1727		
795	0.4431	0.5570		5	0.0304	6.086	10.6
700	0.1375	0.1950	0.191	95	0.3056		
695	0.1302	0.1870		5	0.0073	1.4625	7.6
600	0.0410	0.0680		95	0.0892		
595	0.0380	0.0640	0.066	5	0.0030	0.6120	9.3
500	0.0030	0.0060		95	0.0350		

For instance, according to the distillation test given in Table 17, we have

$$\begin{array}{ll} Q_0 = 1000, & Q = 500, \\ s_0 = 7.72, & s = 0.006, \end{array}$$

hence

$$\left(\frac{1000}{500}\right)^{k-1} = \frac{7.720}{0.006},$$

$$k - 1 = \frac{\log_e 7.72 - \log_e 0.006}{\log_e 2} = 10.3,$$

$$k = 11.3.$$

This value of k , applied to distillation operations where the concentration of the ammonia liquor of the quality given above varies from 7.72 to 0.006 g. per liter, will give results corresponding with those actually obtained."

In chemical engineering work, it will be appreciated, there are a large number of factors of a more or less indeterminate character which arise in connection with almost every problem, rendering a precise solution difficult.

STILL AND DISSOCIATOR

In this book the word "still" is used in its ordinary acceptation, and not as representing the author's view of the correct term to be applied to a plant or apparatus in which the disengagement of ammonia is effected. The term "dissociator" would doubtless be more accurate. This latter appellation has been applied to the apparatus in which the decarbonation and desulphurisation of ammonia liquor are effected by the application of heat in the production of caustic ammonia. Here one has to deal with chemical affinity and physical solution on the counter-current principle.

The ammonia, hydrogen sulphide, and carbon dioxide present in gas liquor are in chemical combination. Hence their partial pressures are appreciably less than when they exist in the free state. The application of heat to about 80° C. results in partial disunion. The three gases previously named, depending on initial concentration, then largely exist in solution—not as hydrosulphide, sulphide, bicarbonate, and carbonate, but, in conformity to Henry's law, as free gases soluble in water under equal conditions.

The solubility of ammonia under like conditions is appreciably greater than hydrogen sulphide and carbon dioxide. Thus the latter gases are dispersed, while ammonia is held in solution.

As, however, the less soluble gases are released at a temperature at which water has a comparatively high vapour pressure, a considerable volume of water vapour is removed, together with a small volume of ammonia in solution. Expressed in another way, there is equilibrium between the tendency for the water to retain ammonia, and the water vapour and acid gases to withdraw ammonia under conditions of temperature and pressure obtaining during the release.

SUPERHEATED STEAM

The question is occasionally asked: Is there any advantage by the use of superheated steam in the distillation of gas liquor?

The following observations on this subject appeared in the 57th Annual Report of the Chief Alkali Inspector:

"Sulphate of Ammonia—important progress in manufacture has been established by Alexander Yuill, the engineer of the Dundee Gas Works, who now supplies to his stills steam superheated.

"Steam is used as the most convenient vehicle of heat; water accompanying adds unnecessary volume of waste liquor (the disposal of which is already difficult enough), and its dilution of the saturator liquor causes delay. Wet steam is a frequent ill at these plants, and for long I have looked forward to its cure.

"Yuill employs a Sugden preheater, fired with coke. He reports (1) that the yield of sulphate of ammonia has increased from 45 cwt. daily to 75 cwt., *i.e.* by 66 per cent; owing to the increased amount of raw liquor he is now able to pass through his stills. (2) Previously wet steam at 28 lb. pressure was used with constant and wasteful drip of water from the drain cock; now the pressure of the dry steam is 22 lb. and its temperature 355-400° F. (3) The superheater has a capacity of 4100 lb. per hour, and supplies steam also to drive tools of the machine shop, so that the chemical plant consumes only a portion of the 10 cwt. of coke used for daily firing. The furnace is cleaned once daily."

The advantage of the use of superheated steam in a still worked at ordinary pressure lies in the reduction of the effluent liquor due to condensation, the superheating of the steam effecting the distillation with a lower condensation constant, and hence rendering unnecessary the employment of supplementary heat. Beyond this there is no material gain over steam applied at ordinary pressure.

Unfortunately, with many small and medium-sized distillation plants, where gas liquor is treated in the manufacture of sulphate of ammonia, there appears to be neglect in the production of dry steam.

Apart from imperfect insulation of the steam mains and pipe lines, it is feared that priming is somewhat prolific.

If attention were directed to the two foregoing points, it is the author's belief that there would be little, if any, advantage to be derived by the use of superheated steam.

EXPERIMENTAL DETERMINATIONS

Fig. 21 represents a complete miniature sulphate of ammonia plant, in which certain experiments have been conducted under varying conditions, with a view to elucidating certain problems bearing upon the general subject of the dissociation of ammoniacal liquors.

A diagram showing the liquor and gas concentrations at two rates of feed, with identical steam consumption, is given in fig. 22. It will be seen from this diagram that the rate of flow has been increased by about 250 per cent at the sacrifice of

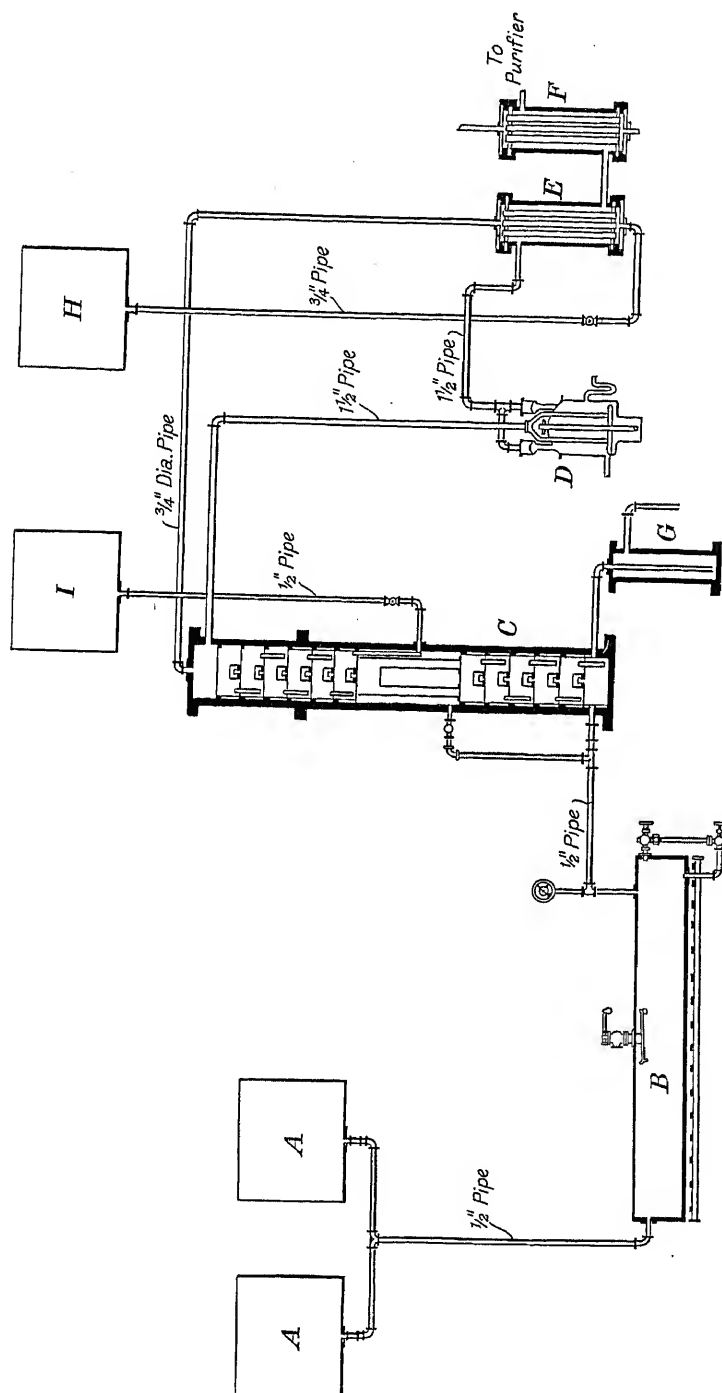


FIG. 21.—EXPERIMENTAL SULPHATE PLANT.

0.23 per cent of free ammonia in the effluent. It is not inconceivable that an identical effluent to that of the lower rate of feed would have been obtained had the time contact been increased, say, to the extent represented by two additional trays.

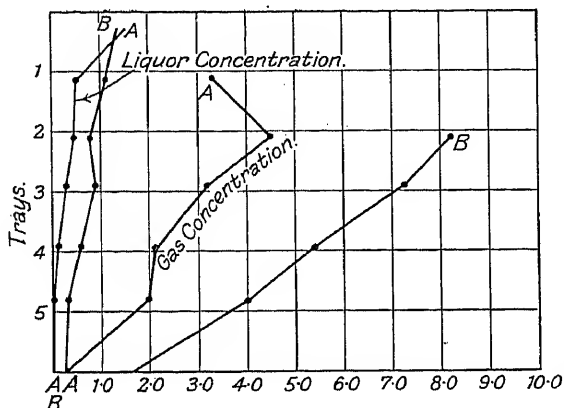


FIG. 22.—GRAPH SHOWING LIQUOR AND AMMONIA GAS CONCENTRATION AT TWO RATES OF SPEED, WITH IDENTICAL STEAM CONSUMPTION.

A, 3.7 gallons per hour; B, 9.3 gallons per hour.

volume of the spent liquor, as contrasted with the gas liquor-devil liquor fed to the still. A simple method of determining this factor under practical working conditions is to condense the stream of steam and ammonia leading to the saturator, and to make a determination for ammonia, carbonic acid, and hydrogen sulphide contents. From a series of analyses carried out in this manner the graph shown in fig. 23 has been prepared. By calculation from the foregoing graph the curve in fig. 24, which affords an indication of the steam consumption per lb. of ammonia in relation to the ammonia concentration in the crude liquor, has been constructed.

Unfortunately, little information appears to be available concerning the extent to which the concentration of ammonia in the gas stream is affected by varying

concentrations of ammonia in the gas liquor. This obviously has an important bearing when fixing the price of gas liquor according to its ammonia content, and particularly so when purchasing concentrated gas liquor. As far as is known, the only recent work published on this aspect is the article by Piron, to which reference has already been made in this chapter. Piron has shown the lines along which problems of this character can be studied, both experimentally and mathematically.

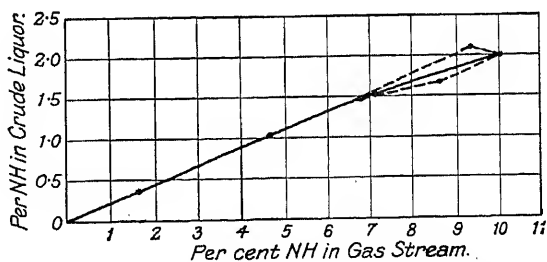


FIG. 23.—GRAPH SHOWING AMMONIA CONCENTRATION OF GAS WITH VARYING CONTENTS OF AMMONIA.

Indeed, in addition to quoting in detail that portion of the article which bears upon the distillation of ammoniacal liquor, it has been deemed desirable to prepare diagrams based on the experimental data afforded by Piron. Fig. 25 is a graph which shows the relationship between the composition of the liquor being distilled and that of the distillate leaving the still. This particular experiment comes under the category of a discontinuous distillation, but the data so given can be correlated to the performance of a continuous still. The graph, in conjunction with fig. 26, which is compiled from data by the same author, is of importance as indicating the economic limit which should be looked for as representing the point at which further distillation is unprofitable. It also furnishes some idea as to the relative value of gas liquor of different concentrations from the point of view of the distiller.

It has been suspected by workers having technical and practical experience of

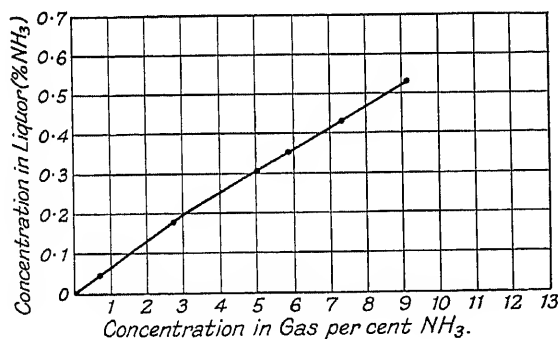


FIG. 25.—DIAGRAM OF EMIL PIRON'S VALUES, SHOWING THE VARIATION OF "K" WITH A VARIATION OF THE STRENGTH OF LIQUOR.

Experiments have been directed to the elucidation of the above view by varying the depth of the seal for a given still design, and although the author is reluctant to draw premature conclusions, evidences are not wanting which support the theory advanced above.

As uncertainty existed as to the intrinsic capacity of a still for dealing with gas

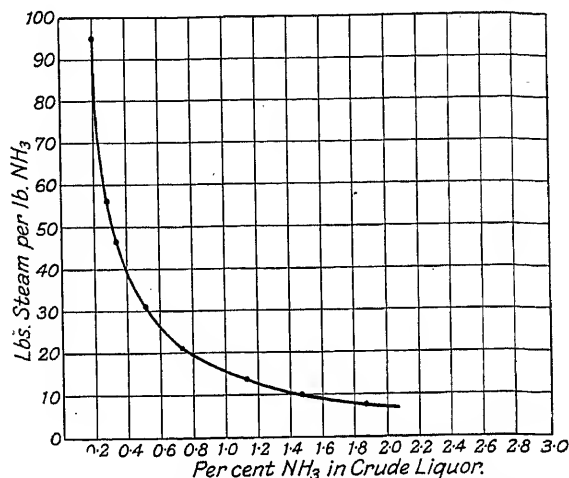


FIG. 24.—CURVE SHOWING STEAM CONSUMPTION PER LB. OF AMMONIA WITH VARYING CONCENTRATIONS OF AMMONIA IN GAS LIQUOR.

gas liquor stills that there is no particular merit in the elements of the still being arranged with a deep seal, through which the ascending gases and steam are compelled to bubble. On the contrary, it has been suggested that the capacity of a gas liquor still can be fully maintained, and the steam consumption appreciably reduced if the ascending gases and steam emerge on the liquor line in the trays. This aspect is enlarged upon in Chapter VI., in dealing with the Hills's still.

liquor of varying concentrations, experiments were made to determine this aspect. Gas liquor of varying concentrations was fed to the experimental still under such

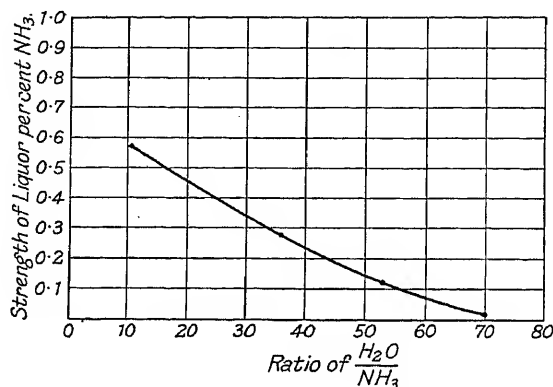


FIG. 26.—STEAM CONSUMPTION PER UNIT OF AMMONIA DISTILLED FROM LIQUORS OF VARYING CONCENTRATIONS, PLOTTED FROM DATA BY EMIL PIRON.

conditions that the effluent liquor leaving the still had approximately a constant composition. From the rates of flow and the concentration fig. 27 has been con-

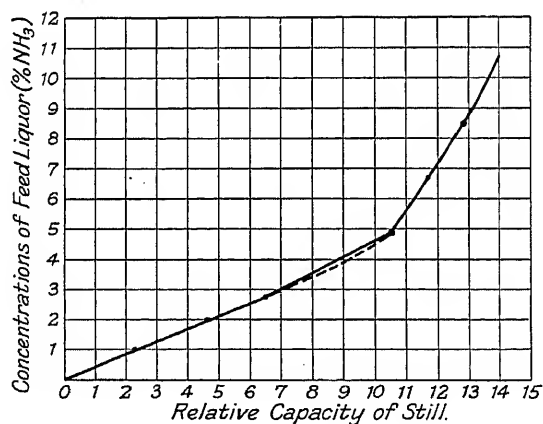


FIG. 27.—DIAGRAM OF THE CAPACITY OF EXPERIMENTAL STILL IN RELATION TO STRENGTH OF FEED LIQUOR.

structed. In brief, this curve represents the amount of ammonia distilled in unit time expressed in relation to the concentration of the feed liquor.

CHAPTER V

INTERMITTENT AND CONTINUOUS DISTILLATION

AN interesting paper which directed attention to the pros and cons of intermittent distillation of ammoniacal liquor was read by J. Wilkinson, F.C.S. (then of Driglington, now Engineer and General Manager of the Nottingham Corporation Gas Works), about twenty-seven years ago. (See *Journal of Gas Lighting*, March 2, 1897.) It is true, as Wilkinson pointed out, that in those days each system had its advocates. It would appear from the discussion which followed the reading of the paper that the fundamental principles involved were not fully appreciated.

It will be well to examine the arrangement of plant which Wilkinson described, and to determine the relative advantages and disadvantages.

Fig. 28 represents the plant. It will facilitate a consideration if the various aspects are dealt with under headings.

INTERMITTENT DISTILLATION

(a) *Simplicity of Operation*

This is the chief merit which the discontinuous process possesses, and one can appreciate that at small works it is a potent consideration in view of the tolerably foolproof character of the plant.

(b) *Fuel Consumption*

One disadvantage of the intermittent process is the inability to take advantage of the available heat of the waste gases leaving the saturator, for preheating the gas liquor.

A further drawback resides in the peculiarly inefficient form of still adopted, chiefly by reason of the disabilities inherent in the setting, and the absence of adequate surface area for heat transmission.

Rispler found that he could obtain an evaporation of only $2\frac{1}{2}$ lb. of water per lb. of coal when he boiled water in an ordinary pot still.

H. M. Lowe, M.Sc., reveals the defects of the pot still and its setting in an admirably lucid paper on the "Theoretical Heat Balance of Tar Distillation." (*Vide Gas World*, Coking Section, January 6, 1923.)

At Driglington the fuel consumption was 12 cwt. of coke per ton of sulphate of ammonia made, using 10 oz. (2.13 per cent NH_3) liquor. The consumption of steam on a continuous plant for similar liquor would be about 25 lb. per 100 lb. of liquor distilled. This figure disregards the heat recovered in the boiler feed water *via* the waste liquor. Taking the sulphate of ammonia as containing 24.50 per cent NH_3 , the following figures are arrived at as representing the quantity of liquor to be distilled in the continuous plant.

The figures are based on a 95 per cent efficiency.

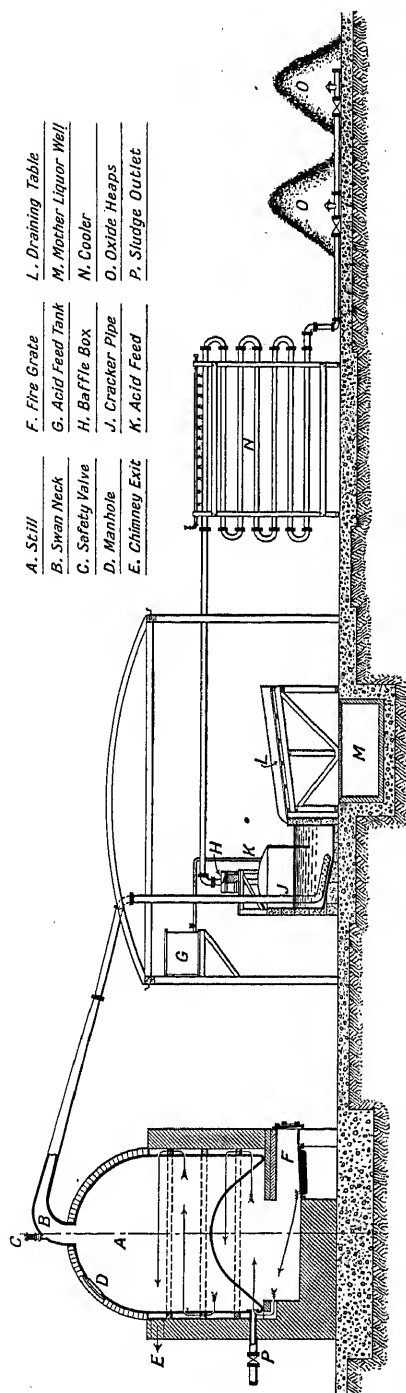


FIG. 28.—INTERMITTENT DISTILLATION PLANT.

$$\frac{24.5}{2.13} \times \frac{224}{1} \times \frac{100}{95} = 2712 \text{ gallons.}$$

The following calculation shows that the consumption of coke per ton of sulphate of ammonia made will be 8.87 cwt.

$$\begin{aligned} & 2712 \text{ gallons} \times 10.25 \text{ (sp. gr.)} \\ & \times \frac{25 \text{ lb. steam}}{100 \text{ lb. of liquor}} \\ & \times \frac{1}{7 \text{ lb. water vapour per lb. of coke}} \\ & = 993.25 \text{ lb., (say) 8.87 cwt.} \end{aligned}$$

It is true that a slight loss of steam may occur between the steam generation plant and the still, but with efficient arrangements suitably controlled this loss should be very slight. Indeed, a consumption of 8.87 cwt. of coke per ton of sulphate of ammonia made may be regarded as a reasonable figure to base upon for the distillation operation alone. There is therefore a saving of 3.13 cwt. of coke in favour of continuous distillation.

(c) Elimination of Ammonia

With batch distillation rarely is the waste liquor reduced below 0.07 per cent ammonia, although in this connection it must be remembered that there is a reduction in the bulk of the liquor with intermittent working as contrasted with appreciable dilution in the case of continuous distillation. Hence it is no criterion to compare the relative ammonia contents in the two cases.

(d) Absence of Uniformity in the Release of the Waste Gases

One disadvantage of the intermittent system is that the release of H_2S and CO_2 from the gas liquor is irregular, and this imposes additional work on the purification section of the plant.

(e) Installation Costs

Installation costs are less with the intermittent system than with the continuous one.

CONTINUOUS STILLS

The chief advantages of continuous distillation are :

- (a) High rate of working.
- (b) Possibility of interchange heating at several points.
- (c) Regularity of operation.
- (d) Reduced fuel consumption.

The disadvantages are :—

- (a) Stills are more complicated in construction.
- (b) More expensive to install.
- (c) Need fairly skilled labour for their operation.

Generally speaking, however, these disadvantages are more than counter-balanced by the advantages enumerated, and to-day few engineers would adopt the intermittent system except for very small works.

WORKING DETAILS FOR INTERMITTENT PLANT

It is interesting to furnish a few working details concerning the intermittent distillation plant illustrated in fig. 28.

The writer is indebted to F. Scholefield, Secretary and Manager of the Driglington and Gildersome Gas Light Co. for some of the details.

The still holds nine tons of liquor and is worked during the day. The fire is commenced at 10 P.M. and attended to during the night by the foreman stoker. The sulphate man proper starts his duties at 5 A.M., and has removed all the salt from the saturator by 3 P.M. The still operates for practically 18 hours, as the fire is not drawn before 5 P.M., shortly after which the spent liquor is discharged. No live steam is used. The first mixing of milk of lime is added about 8 A.M. and then at regular intervals till 1 o'clock. No trouble has been experienced with the burning of the plates. The still has been in operation for twenty years. The saturator temperature is satisfactory, and the salt dries without much difficulty.

It will be appreciated that a more efficient type of still for intermittent working could be adopted than the ordinary pot still.

A Lancashire or Cornish boiler so seated as to promote the best possible radiative power of the products of combustion, and to secure maximum surface for heat transmission, would constitute a more efficient apparatus, but would, of course, handicap the ready discharge of the spent liquor, and the recharging of gas liquor.

The realisation of the expense involved in working direct fired stills apparently led to the adoption of plant as shown in fig. 29. This arrangement comprises a combination of three boilers so disposed that the volatile compounds and steam expelled from one of the lower boilers pass to the top one. The lower boilers are worked alternately. When sufficient liquor has been accumulated in one, the necessary milk of lime is added and the steam is passed through the closed coil. Meanwhile the waste liquor is run off from the second lower boiler preparatory to receiving more liquor from the continuously operated higher boiler.

A record appears in Muspratt's *Chemistry* to the effect that in 1841 the Coffey

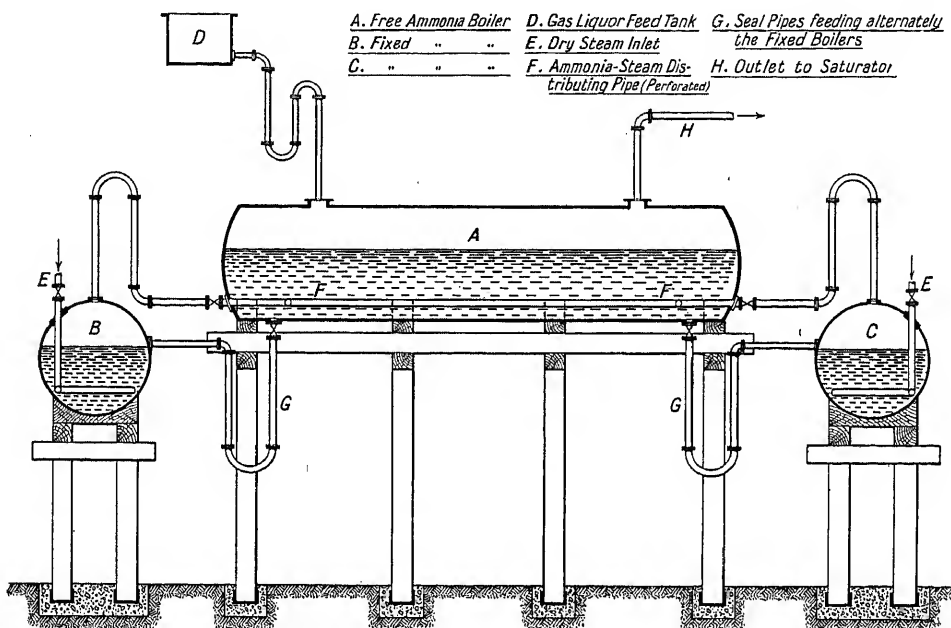


FIG. 29.—COMBINATION TYPE OF BOILER STILL.

still was adapted by Newton & Co. to gas liquor distillation. This appears to be the earliest record of continuous ammonia distillation.

Subsequently Savalle was responsible for the introduction of a continuous still, on which most of the present-day apparatus is based.

Fig. 30 shows a portion of Savalle's still. It comprises a vertical column divided horizontally into a number of chambers. The liquid to be distilled descends chamber after chamber by means of the overflow pipes "C."

A strong current of steam is introduced at the bottom of the column, and ascends through the perforated caps "B."

This aids distribution of the steam through the liquor flowing through each chamber. As all the liquid passes in turn through each of the compartments, clearly by the time it reaches the bottom the volatile constituents must have been

expelled. Fresh liquor enters the top column, meeting the steam which already contains large quantities of ammonia. Thus an effective counter-current is established, the liquor having the greatest content of ammonia coming in contact with the richest ammonia-containing vapours, and steam is applied to the liquor which is practically denuded of ammonia.

Obviously an apparatus directed to bring small and regular quantities of liquor in contact with a counter-current flow of steam, admitting of large wetted surfaces and ensuring efficient distribution of the steam through the surface of the liquor, must constitute an ideal arrangement which the best type of boiler still, or combination of continuous working batch stills cannot approach in point of efficiency.

As illustrating the most advantageous method of applying heat in the distillation of ammonia, the following results of experiments carried out by Dr. C. M. Tidy on a manufacturing scale, dealing with 7000 gallons of liquor, are afforded :

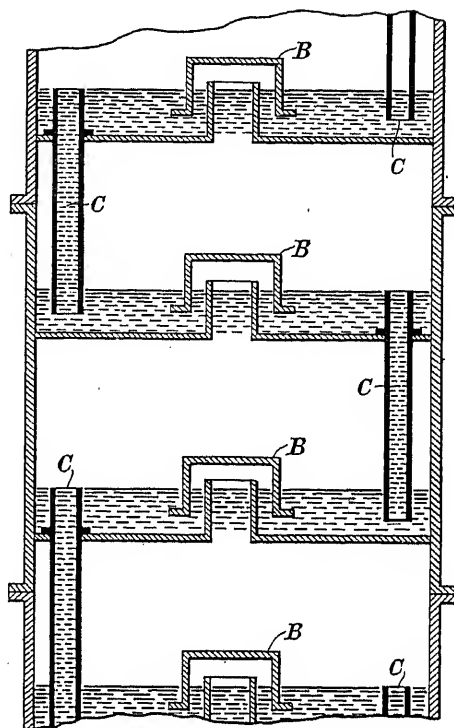


FIG. 30.—SECTION OF SAVALLE STILL.

TABLE 18

	No. of Hours.	Efficiency of Expul- sion of Ammonia.
		Per cent.
When heated by direct fire	22	90
When heated by a closed steam coil . .	18	92
When heated by open steam (wet) . .	14	98.5

These results demonstrate the advantage of direct steam.

STILLS FOR SMALL WORKS

It not infrequently happens that even smaller discontinuous stills than 9 to 10 tons capacity per day are required. One can appreciate that a small works, carbonising (say) two thousand tons of coal per annum and producing something of the order

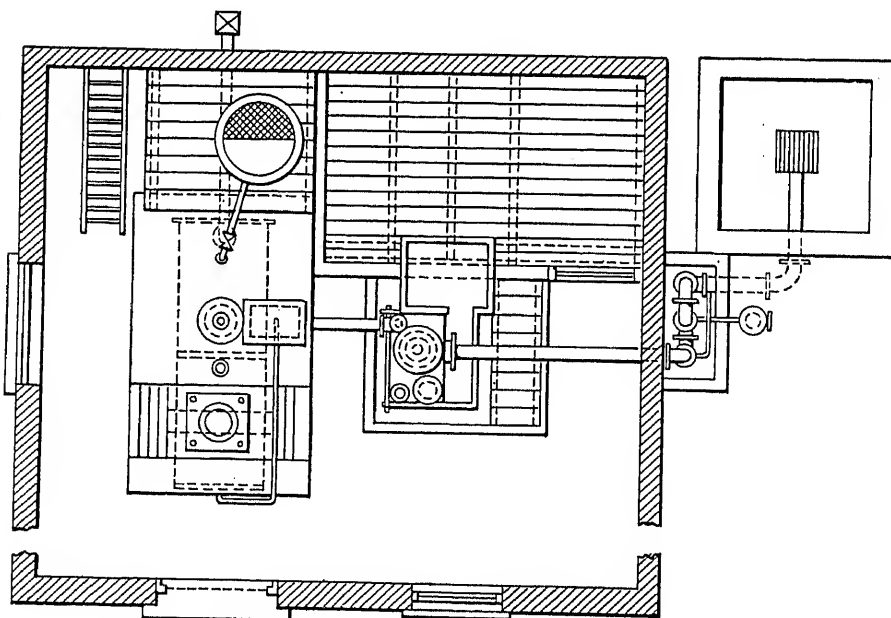
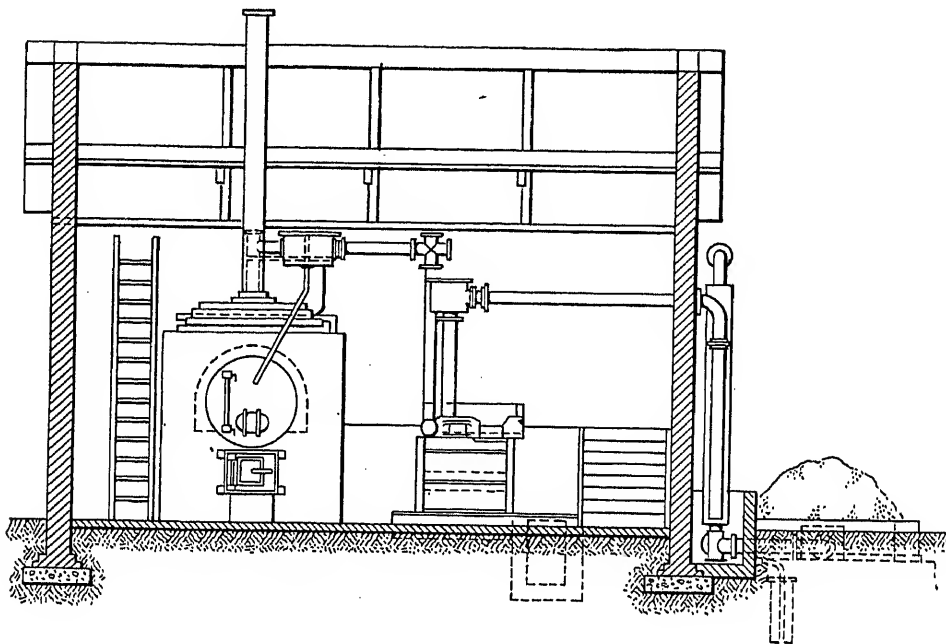


FIG. 31.—WALKER'S INTERMITTENT SULPHATE PLANT FOR SMALL WORKS.

of 70,000 gallons of 8 oz. liquor per year, may find it necessary to produce sulphate of ammonia, if the costs of transport of the liquor actually represent more than the value of the liquor at the gas works. Several such cases have come to the notice of the writer. Gas managers in these cases are confronted with two considerations: Is it more profitable to make sulphate of ammonia in a suitable small discontinuous plant, or to concentrate the gas liquor so as to minimise transport charges?

Local circumstances and special considerations invariably afford an answer. In any case, the decision reached in not a few cases has led to the installation of a small sulphate of ammonia plant, forming part of which is a still usually 9 feet long by 3 feet diameter, seated in brickwork, and having a suitable hearth, and necessary equipment for the combustion of fuel, together with a small steel chimney for the withdrawal of the products of combustion. The still is provided with a gauge glass, McNeill manlid, and outlet pipe and cock, and the pipe conveying the ammonia-laden vapours leads into a D-shape baffle box, so as to arrest any entrained liquor. The liquor which is arrested is returned to the still.

Such a still and setting, together with all the necessary equipment to constitute a self-contained sulphate of ammonia plant, is supplied by C. & W. Walker, Ltd., of Donnington, Salop. The merits of such a plant, which is shown in fig. 31, are its simplicity and foolproof character. An intelligent labourer should have no difficulty in operating such a plant. Of course, in these days of reduced prices for sulphate of ammonia, and high costs of production, the margin of profit in connection with such a plant will not be appreciable. Nevertheless, in those cases where the actual removal of the liquor from the gas works has constituted a charge, one can conceive of the necessity for the installation of such a plant.

CHAPTER VI

THE DESIGN OF AMMONIA STILLS

ALTHOUGH it is possible to trace a patent by Watson for an ammonia still as early as 1838, and the application of "Coffey's still with a few modifications to adapt it to the distillation of gas water" by Newton in 1841, a quest for literature relating to the design of ammonia stills is singularly ineffectual.

The only conclusion to be reached is that the subject is essentially a new one. It is, however, replete of fascinating problems.

The origin of the present type of continuous columnar still for the distillation of gas liquor can be traced to a Frenchman of the name of Savalle. He was responsible in 1875 for many patents directed to the improvement of the apparatus and processes for the distillation, purification, and rectification of alcohol.

Savalle's inventive genius was later turned to an improvement of the continuous columnar still for the distillation of ammoniacal liquor. Here is an instance not uncommon in the chemical industry, where the experience and practice of one industry, due to seemingly analogous conditions, is applied to the service of another.

TYPES OF AMMONIA STILL

Broadly speaking, the arrangement of the stills and liming chamber can be divided into three types:

- (a) Free still superposed on liming chamber, with fixed still separate;
- (b) Free still superposed on liming chamber, with fixed still; and
- (c) Free still superposed on fixed still, with liming chamber separate.

These types are illustrated in fig. 32. As to the arrangement which the plant should

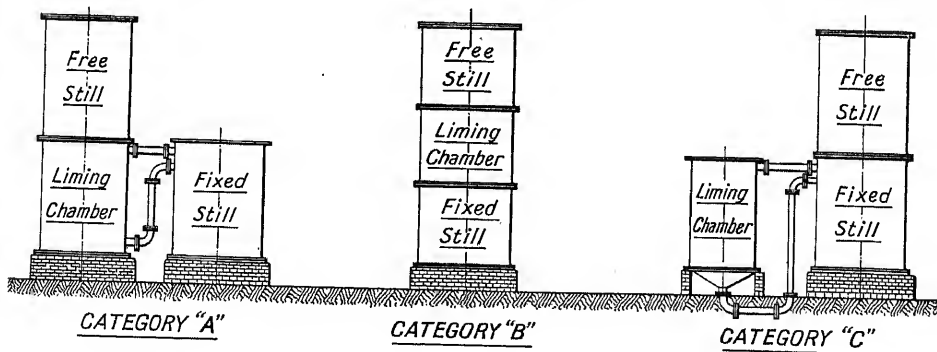


FIG. 32.—ARRANGEMENT OF DISTILLATION PLANT IN THREE CATEGORIES.

take, much depends on the provision made for cleaning the stills, on the fixed ammonia content of the gas liquor to be distilled, and on its freedom from tarry matter.

SOME FEATURES OF AMMONIA STILLS

By far the greater number of stills operating in the United Kingdom are stills built up of double-flanged sections, each of which, generally speaking, constitutes a

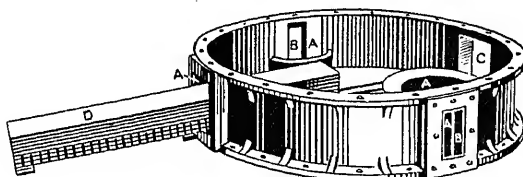


FIG. 33.—CLEANING ARRANGEMENT (WILTON).

chamber. The joints between the corresponding flanges of the chambers are made of boiled linseed oil and Vulcan cement, or with red and white lead, and lead wire.

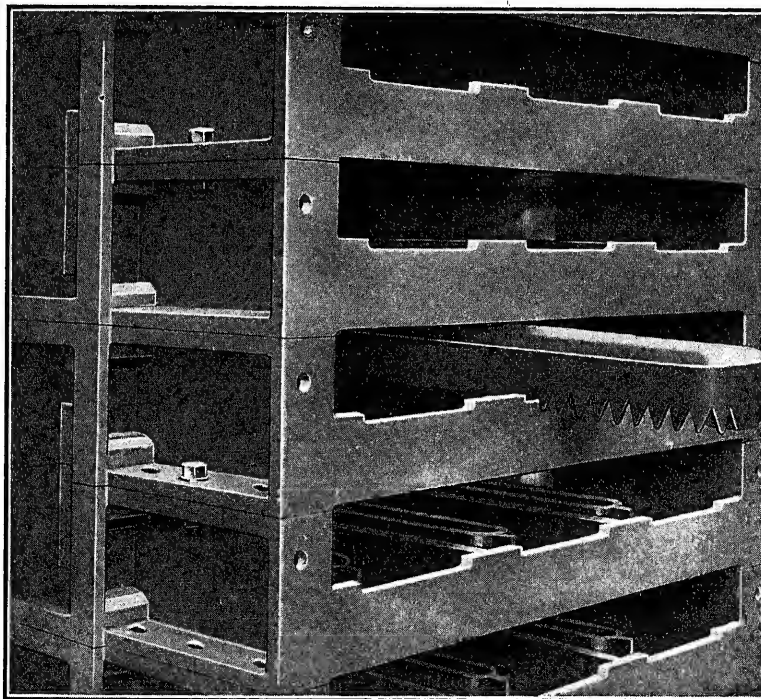


FIG. 34.—CLEANING ARRANGEMENT OF TRAYS (HOLMES'S STILL).

Obviously, these sections have to be provided with such a number of manholes, and of such a size, as will afford facilities for the cleaning of the overflow pipe or pipes, and the removal and/or cleansing of the hoods, "frogs," "crocodiles," or whatever

vapour-distributing device is provided. Figs. 33, 34, and 35 show respectively the special cleaning arrangement provided in the stills as made by the Chemical Engineering & Wilton's Patent Furnace Co., Ltd., W. C. Holmes & Co., Ltd., and C. & W. Walker, Ltd. In the sectionally constituted stills the several joints are potential sources for the leakage of ammonia, and as it is unusual to insulate the covers of the manholes with non-conducting material, the loss of heat by radiation is not inconsiderable.

Complete dismantling may become necessary, owing to the accumulation of lime sludge, either causing blockages—which are difficult, if not impossible, to remove *in situ*—or the lime may form laminations on the bottoms of the chambers to such an extent as seriously to impair the thermal efficiency and capacity of the plant. Another

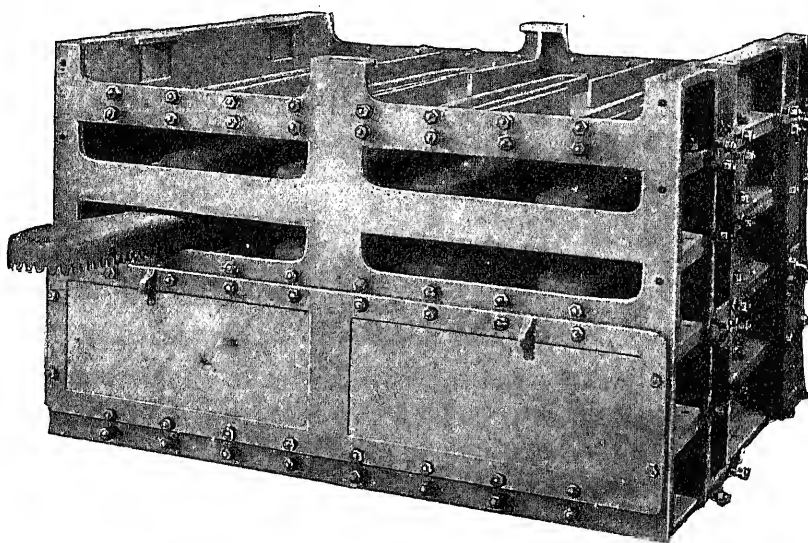


FIG. 35.—CLEANING ARRANGEMENT OF TRAYS (WALKER'S STILL).

arrangement of still is possible, which largely overcomes the disabilities to which reference has been made.

Fig. 36 represents the alternative form, which comprises an outside shell free from a multiplicity of flanges, and without cleaning manholes. This design of still, for which Dr. Carpenter was responsible, contemplates the withdrawal of the internal elements, which, as shown in figs. 37 and 38, comprise a hood, tray, and bubbler, as constituting a complete chamber, for cleaning purposes.

These hoods, trays, and bubblers are provided with machined faces, so that there is no possibility of the by-passing of steam on the outer part of the trays. Suitable lifting tackle, as seen in fig. 39, is provided for facilitating the removal of the internal elements, which, in turn, are all provided with suitable lifting lugs.

It has been found in practice that, provided the accumulations of lime sludge at the foot of the liming chamber are removed every fortnight, and the top cover of the

fixed still is lifted monthly, for the cleaning of the perforations of the anti-priming device, the still will operate continuously for six months before cleaning is necessary. During this time approximately 5,000,000 gallons of gas liquor will have been distilled, still cleaning representing a charge of about 2d. per ton of sulphate made. With the shell type of still, involving as it does the withdrawal of the internal elements

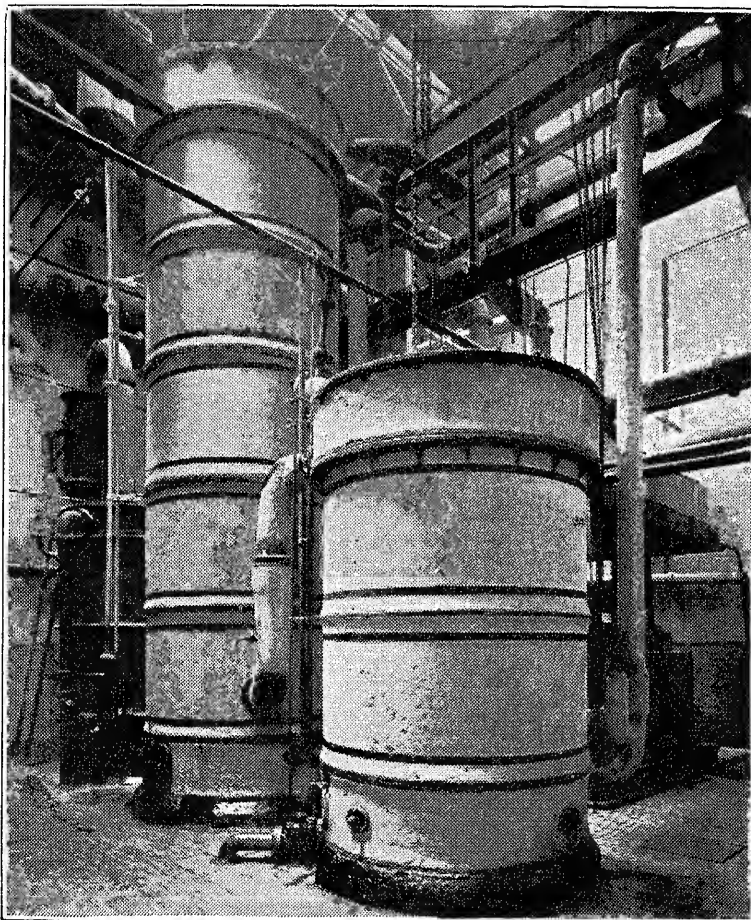


FIG. 36.—AMMONIACAL LIQUOR STILL (DR. CARPENTER'S DESIGN).

for cleaning purposes, it would obviously be unwise to arrange the plant according to category "B" (see fig. 32), as this would necessitate the removal of the liming chamber in order to afford access to the anti-priming arrangement, or first tray, of the fixed still.

Fig. 40 represents a gas liquor still as installed at the Provan Chemical Works of the Glasgow Corporation. The fixed ammonia content of the gas liquor in this case only represents about 10 per cent of the total amount.

Manifestly with gas liquor having a high content of fixed ammonia, particularly if such fixed ammonia is due to ammonium sulphate, the difficulty of keeping the

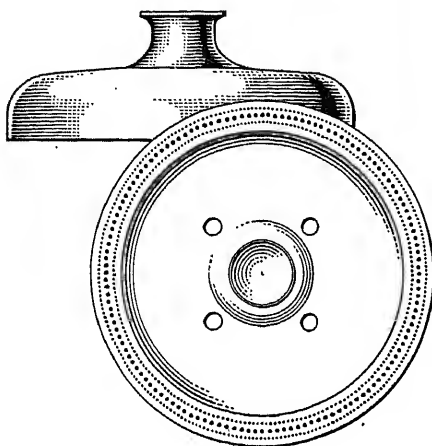


FIG. 37.—HOOD AND BUBBLER.

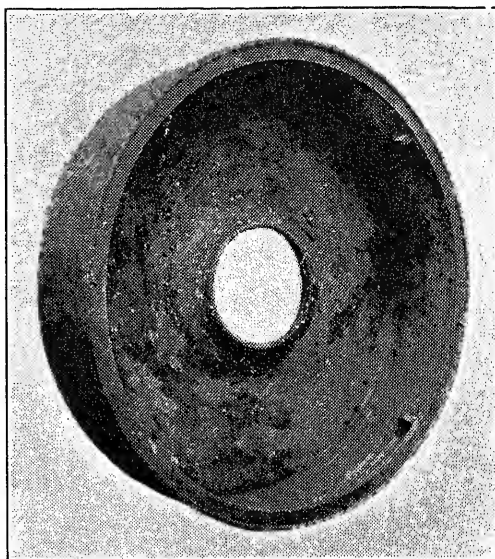


FIG. 38.—TRAY.

distilling sections of the fixed still free from sludge, particularly the first and second sections, is great. Special consideration should be given to the design of the liming

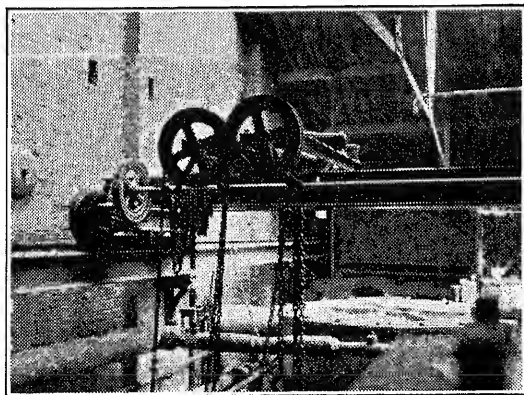


FIG. 39.—LIFTING TACKLE.

chambers, or the elements of large liming chambers, which admit of considerable time contact, in order to ensure active agitation. Easy means of a periodic discharge of the sludge from the foot of the liming chambers should also be provided. Fig. 41 shows a typical provision in this respect. Failing this, an arrangement of plant with outside liming section specially designed as represented in category "C" is necessary.

It is the author's view that mechanical considerations (such as facilities for cleaning, provision for

the removal of bubbling hoods) and initial capital cost have been the factors largely governing the design and arrangement of gas liquor stills in the past. Had designers and makers of gas liquor stills been in a position to visualise the

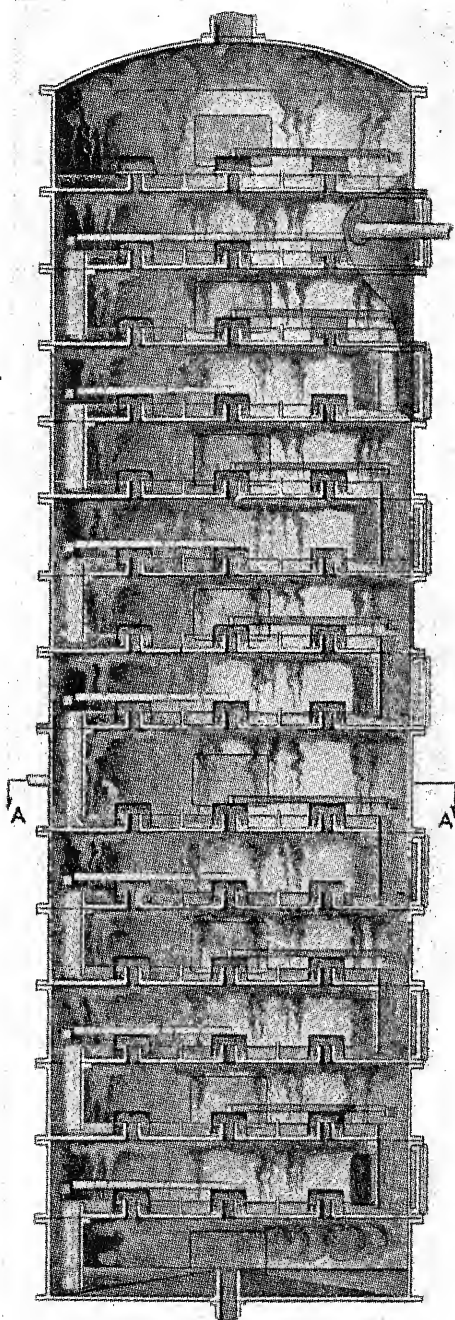
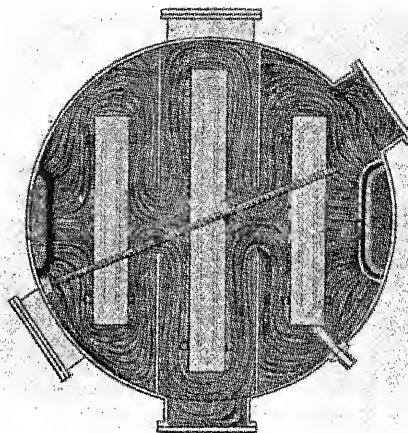


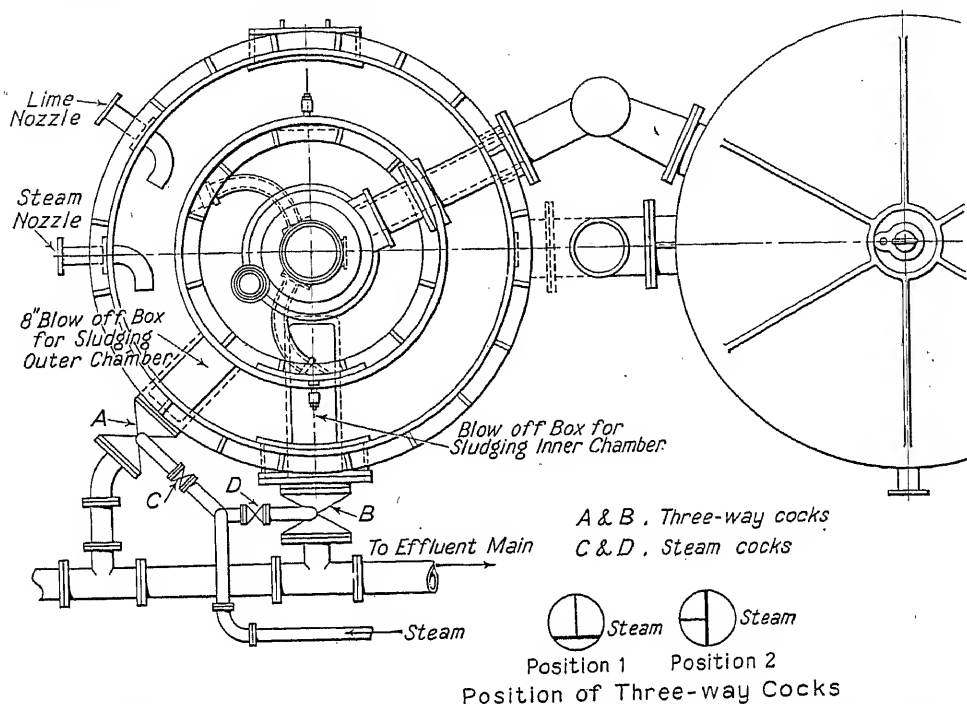
FIG. 40.—AMMONIACAL LIQUOR STILL
(PROVAN—W. A. WALMSLEY, B.Sc., MANAGER).



items which contribute to the cost of ammonium sulphate in so far as the still itself is concerned, it is not improbable that less attention would have been directed to this somewhat circumscribed aspect of design, and more attention given to the possibilities of designing a still which would be economic as regards steam consumption. In saying this, it is not overlooked that the ideals of the chemist and physicist have not infrequently to be compromised in order to ensure mechanical strength and practicability in the matter of production.

The data given in Table 19 relative to the characteristics of several types of present-day stills are impressive in many ways, and will repay careful study. While recognising the various local conditions under which stills have to operate, and the variation of the quality of liquor with which they will be called upon to deal, one is led almost irresistibly to the conclusion that standardisation based on experimental data under varying conditions, and with due regard to the fundamental laws governing the distilla-

tion of gas liquor, would be fully warranted. Table 19 shows clearly the confusion



To sludge down the outer chamber, set cock A to position 1, opening steam cock C. After agitating the deposited sludge, cock A is set to position 2, cock C closed, and the sludge discharged to the effluent main. A similar procedure takes place for the inner chamber except cock B is set to position 1, steam cock D being opened. After satisfactory agitation cock B is set to position 2 and cock D closed, the sludge being discharged to the effluent main.

FIG. 41.—AMMONIACAL LIQUOR STILL (SLUDGING ARRANGEMENT).

of types and dimensions which has arisen, and the obvious inference is that all these stills cannot alike be efficient and economical.

THE IDEAL STILL

The ideal still would be one of such a height beyond which there would be no increase of ammonia concentration in the steam-ammonia stream. Such a still would involve a minimum steam consumption, and would ensure complete, or practically complete, elimination of the ammonia from the outflowing waste liquor.

Of course, when one is surrounded by mundane considerations, a still of the contemplated height is out of the question from the monetary point of view. Moreover, it must be remembered that so far as maximum ammonia concentration is concerned, this is governed by the temperature of the in-going liquor, the partial pressure of which is a function of the temperature to which it has been preheated. It is known from practical experience that the quantity of steam applied to remove the last traces of ammonia may be out of all proportion to the value of the infinitesimal quantity of ammonia released. There is therefore a limit to the use of steam in relation to the amount of ammonia expelled.

TABLE 19

CHARACTERISTICS OF AMMONIACAL LIQUOR STILL AS SUPPLIED BY ENGLISH MAKERS

Letter Index.	Dia- meter of Free Still.	Height of Fixed Still.	Diameter of Fixed Still.	Height of Fixed Still.	No. of Trays in Free Still.	No. of Trays in Fixed Still.	Depth of Liquor in Trays. (galls.)	Liquor Capacity in Trays. (galls.)	Depth of Liquor Seal.	Heat- ing Surface Tray or Ele- ment.	Capa- city per Hour (galls.)	Lb. of NH ₃ per Hour. (2% NH ₃ liquor)	Surface Area occupied by Hoods, Crocodiles or other such Devices.	Depth of Cham- ber.	Arrange- ment of Overflow Device.	Arrange- ment for Cleaning.	Arrange- ment at Top of Still to avoid Entrain- ment.	Surface Area for Descent of Liquor.	Weight of fully equipped Still.	
	ft. in.	ft. in.	ft. in.	ft. in.			in.		in.	sq. ft.	sq. ft.	(2% NH ₃ liquor)	sq. ft.	ft. in.				sq. in.	sq. in.	tons.
A	5 10	21 10	5 10	10 2	6	5	5	57.25	1	21.59	1250	950 (2% NH ₃ liquor)	12.48	1 4½	Annular space round hood	Still to be dis- sembled	Anti- priming device	71.6	278	41
C	4 0	16 2½	4 0	9 11	10	8	3½	17.02	1½	9.08	1050	182	4.9	11	Two semi- elliptical slots	Large manholes	Void	183	224	22
G	7 6	18 8	7 6	11 5	9	8	2	43.44	1	41.7	1300	235 (1.73%)	11.6	1 0½	Eight 4" pipes	Manholes	Void	248	59.5	..
I	5 6 square	17 7	5 6 square	7 0½	12	8	2½	41.2	1½	29.15	3.9	9	One 8" x 3" opening	Hand- holes	Large void	110	48	..
J	5 0	19 8	5 0	8 11	9	8	2½	21.2	1	17.04	5.37	10	Two semi- elliptical slots	Hand- holes	Void	174.7	180	..
L	4 6	7 6½	4 6	7 6½	13	8	1½	12.5	1½	12.54	406	70.2 (1.73%)	6.09	6	Six 4" diameter pipes	Six handholes 6" x 3" for each chamber	Void	217.5	73.75	15
Category "B"																				
B	5 0	12 6	Com- prising a conical base with circular steps in- creasing in dia.	6 8	14	9 steps	1½	12.75	1½	19	650	141 (2.17%)	11	7½	Two 4" pipes	Manholes	Anti- priming hood	78.5	14	25
F	7 0	12 0	7 0	12 0 inclusive of lining chamber	8	6	3½	58.1	1½	32	2910	437.4 (1½%)	11.53	1 6	Two semi- elliptical slots	Large manholes	Void	373	522	19
H	6 0	4 0	6 0	6 6	6	8	2	28.0	1	26.9	840	182.2 (2.17%)	12.2	7½	One 8" x 4" semi-ellip- tical pipe	Hand- holes	Void	113	57	17½
Category "C"																				
D	5 4	9 0	5 4	12 8	5	7	5	49.5	2	18.96	1250	195 (1.56%)	5	1 3	One semi- circular pipe	Manholes	Void	213	204	..
E	5 6 square	4 0	5 6	8 6	4	8	2½	39.3	1½	20.25	560	56 (1%)	11.6	9	12" x 4" openings	Manholes	Void	528	54	..
K	5 6 square	8 0	5 6	13 0	10	15	2½	36.4	1½	28.0	800	126 (1½%)	11.6	9	Four 7" x 3" openings	Manholes	Void	528	84	26

In attempting to visualise the best type of still, having regard to the many considerations involved, it will be seen that a compromise is imperative. If, as would appear from a consideration of cost sheet figures relating to sulphate of ammonia manufacture, the consumption of steam is the dominating consideration, it is evident that the still must be designed with due knowledge of this factor.

The absorption of heat in any still must be as follows :

- (1) The heat required to raise the liquor to boiling-point, at the still pressure (this obviously varies according to the extent to which the liquor is pre-heated) ;
- (2) The dissociation of the ammonium salts, and ammonium hydrate, if present ;
- (3) The radiation losses ; and
- (4) Increase of boiling-point as the liquor descends the still, due to increase in pressure.

HEAT ABSORBED IN THE DISSOCIATION OF AMMONIA FROM ITS SALTS

F. H. Wagner, in his book on *Coal Gas Residuals*, states the heat absorption per lb. of ammonia dissociated in the still from salts as 1880 B.Th.U. Unfortunately, there is little information available on this aspect.

Basing on Thomsen's determination, as furnished in his book on *Thermal Chemistry*, the following figures have been computed :

TABLE 20

<i>Heat of Solution</i>				Calories.
NH ₃ (including hydration)	.	.	.	8.43×10^3
H ₂ S	.	.	.	4.56×10^3
CO ₂	.	.	.	5.88×10^3

Heat of Formation

NH ₃ + H ₂ S + NH ₄ HS Aq.	.	.	.	6.19×10^3
2NH ₃ Aq. + CO ₂ Aq. + (NH ₄) ₂ CO ₃ Aq.	.	.	.	15.90×10^3

Heat of Formation of NH₄HS Aq. in B.Th.U. per lb. NH₃

$$\begin{aligned}
 8430 + 4560 + 6190 &= 19,180 \text{ c.} \\
 19,180 \times 0.00398 &= 76.33 \text{ B.Th.U.} \\
 \text{NH}_3 \text{ Aq.} &\longrightarrow \text{NH}_4\text{HS Aq.} + 76.33 \text{ B.Th.U.} \\
 17 & \\
 17 \text{ gms.} : 453.6 &:: 76.33 : 2037 \text{ B.Th.U.} \\
 &(\text{gms. per lb.})
 \end{aligned}$$

Heat of Formation of $(\text{NH}_4)_2\text{CO}_3$ Aq. in B.Th.U. per lb. NH_3

$$\begin{aligned} (2 \times 8430) + 5880 + 15,900 &= 38,640 \text{ c.} \\ 38,640 \times 0.00398 &= 153.8 \text{ B.Th.U.} \\ 2\text{NH}_3 \text{ Aq.} \longrightarrow (\text{NH}_4)_2\text{CO}_3 \text{ Aq.} + 153.8 \text{ B.Th.U.} \\ 34 & \\ 34 : 453.6 :: 153.8 : 2052 \text{ B.Th.U.} \end{aligned}$$

DISPOSITION OF HEAT AS AFFECTING THE AMMONIA STILL

Turning now to the heat balances to be found in Chapter IX., it is interesting to examine the disposition of the heat as affecting the still itself.

The following figures represent the heat applied to and generated within the still :

TABLE 21

<i>Heat applied</i>	B.Th.U.
1. <i>Via steam</i>	252,400
2. <i>Via preheated liquor (88.2° C.)</i>	134,000
3. <i>Via hot cream of lime (70° C.)</i>	9,300
<i>Heat generated</i>	
4. Heat of reaction between lime and ammonium sulphate to produce 3.2 lb. NH_3 in solution	5,100
	<hr/> 400,800 <hr/>

How is this heat accounted for ? The following figures afford an answer :

TABLE 22

	B.Th.U.
1. To heating 1020 lb. liquor from 88.2° to 105° C.	30,600
2. To heating 95 lb. cream of lime 70° to 105° C.	7,885
3. To dissociation of 12.7 lb. NH_3 from solution of NH_4HS and $(\text{NH}_4)_2\text{CO}_3$	23,960
4. To dissociation of 3.2 lb. NH_3 from aq. solution	2,870
5. To total heat of 178 lb. steam at 16 lb. abs. sensible heat of NH_3 , H_2S and CO_2	201,800
6. To heat loss <i>via radiation</i>	11,700
7. To heat loss <i>via effluent liquor</i>	182,200
	<hr/> 461,015 <hr/>
By subtraction of items 1 and 2 (accounted for in 7)	38,485
<i>Heat accounted for</i>	<hr/> 422,530 <hr/>

It is hoped that the diagrammatic sketch (fig. 42) will enable the reader to visualise more clearly the distribution of the heat referred to above.

It will be noted that more heat is accounted for than is actually applied to or generated within the still.

This discrepancy may possibly be explained by the inclusion of the heat of solution (which may conceivably be again given up to the still), as constituting part of that required for the dissociation of the ammonium compounds, and the balance may be due to a slight inaccuracy in the determinations.

It will be appreciated that it is one thing to be able to explain the distribution

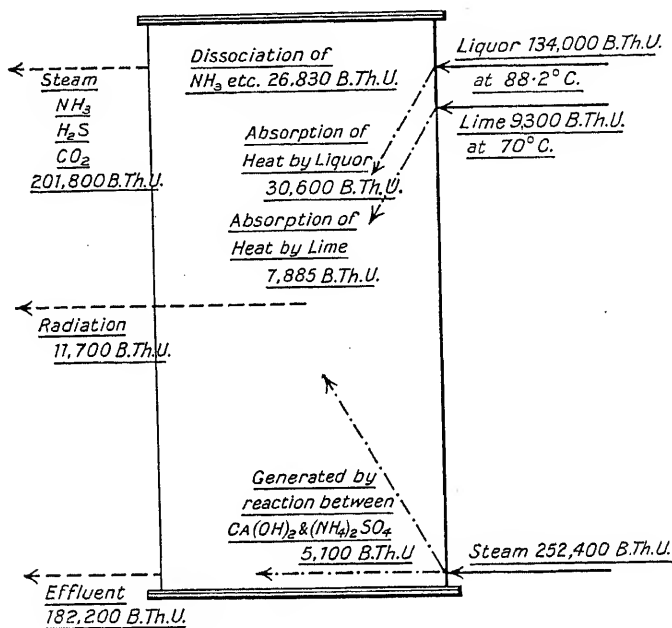


FIG. 42.—DIAGRAM SHOWING DISTRIBUTION OF HEAT IN STILL.

of heat applied to a distillation unit, and another thing to affirm that such heat is necessary. Expressed figuratively, it is easy for a cashier to balance the wages he pays with the money he receives from the bank for such payment, but no cashier can say definitely whether the money paid is an accurate measure of the work done.

In whatever way an attempt is made to check the disposition of heat as affecting the ammonia still, some figure has to be taken into account which represents actual results obtained during the working of the particular still as distinct from ideal achievement.

This submission can best be elucidated by re-examining the heat balance already mentioned from the point of view of the still. The calculations are based on 1 lb. of ammonia dissociated from its compounds, with gas liquor of 1.59 per cent NH_3 content.

TABLE 23

	B.Th.U.	Lb. Steam.	Percentage.
1 lb. of ammonia requires for dissociation	2044	2.12	12.70
61.9 lb. water heated from 88.2° C. to 105° C.	1863	1.94	11.60
6 lb. water (with lime) from 70° to 105° C.	380	0.39	2.30
Radiation loss	800	0.83	5.00
11.4 lb. water in steam-ammonia stream leaving the still	11.40	68.40
	..	16.68	100.00

The actual heat applied to the still was 16.35 lb. of steam per lb. of ammonia dissociated, but the ammonia concentration of the ammonia-steam stream leaving the still, which accounts for 68.40 per cent of the steam required in the dissociation of 1 lb. of ammonia, under the conditions cited, does not actually represent ideal achievement, and in so far as the performance of the still falls short of what is an ideal standard, so far is improvement possible.

What represents ideal achievement? Is it necessary to evaporate 11.4 lb. of water in dissociating 1 lb. of ammonia from gas liquor of 1.59 per cent NH_3 content?

In the present state of our knowledge this question cannot be definitely answered.

HEAT LOST BY RADIATION

In connection with certain figures bearing on this subject, published by the author in a paper read before the Chemical Engineering Group of the Society of Chemical Industry in Glasgow in 1922 (*vide J.S.C.I.*, vol. 41, 229t., July 31, 1922), criticism was levelled against the figure given as representing the loss of heat by radiation from the still.

The stills in question are of the shell type. There are virtually two thicknesses of iron. Insulation is afforded by Leroy's boiler composition to the extent of $1\frac{1}{2}$ in. thick. In connection with the heat balances included in Chapter IX., further determinations have been made, and a slightly different figure has been reached as representing the loss of heat from this source. To confirm the accuracy or otherwise of the figures in question, a separate determination of the radiation loss from a still was made in the following manner:

- (1) The still was filled with water, *i.e.* the bubbling devices and liming chamber were filled to the level of their respective overflows.
- (2) The water was heated by means of steam to 100° C.

- (3) The steam was turned off and the temperature drop observed by means of a recording thermometer, the bulb of which was placed in a central position in the still.
- (4) From the range of temperatures so obtained a curve (see fig. 43) was drawn,

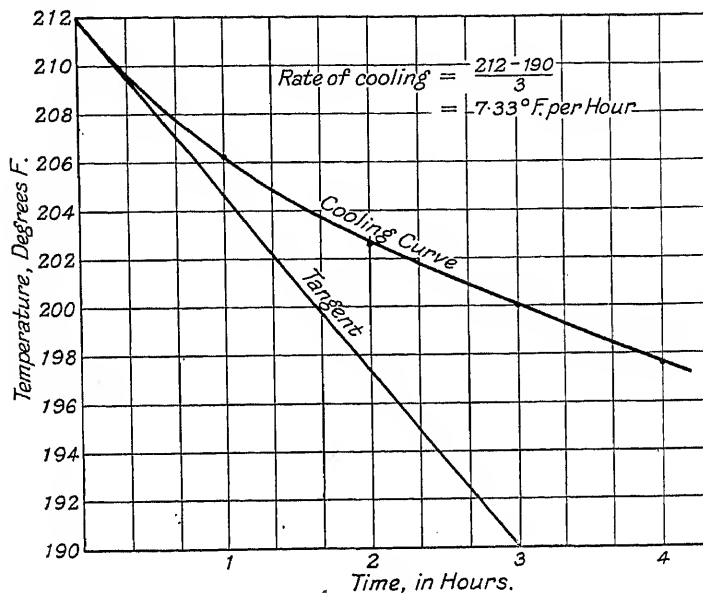


FIG. 43.—COOLING CURVE GRAPH.

plotting degrees Fahrenheit against time in hours. A tangent, drawn to the hottest part of the curve, gave the rate of cooling at 212° F. to be 7.33° F. per hour.

- (5) The weight of water contained in the free and fixed portions of the still was calculated to be 12,174 and 3714 lb. respectively, while the weights of the stills were known to be 58,800 and 35,840 lb.
- (6) The following calculations show the heat loss in B.Th.U. per minute; the specific heat of water being taken as 1.

$$\text{A. Free still : } \frac{12,174 + (58,800 \times 0.13)}{60} \times 7.33 = 2421 \text{ B.Th.U. per min.}$$

$$\text{B. Fixed still : } \frac{3714 + (3584 \times 0.13)}{60} \times 7.33 = 510 \text{ B.Th.U. per min.}$$

$$\text{Total} = \text{A} + \text{B} = 2931 \text{ B.Th.U. per minute.}$$

- (7) The surface area of the stills was calculated to be 667 sq. ft., so that the loss per square foot was $\frac{2931}{667} = 44 \text{ B.Th.U. per minute.}$

The figure arrived at is practically identical with the modified radiation loss included in the heat balances.

FACTORS AFFECTING STILL DESIGN

There are many factors affecting the design of stills, all of which must have some bearing on the consumption of steam required per unit weight of ammonia dissociated. It is proposed to deal with these factors (not necessarily in order of importance, but as they occur to the author) separately.

(1) *Extent of Seal*.—There is no necessity for a deep seal; 1 in. depth of seal is preferable to 4 in. depth. The cumulative effect of a number of deep seals is to render it more difficult to remove the last traces of ammonia. Incidentally, the consumption of steam within the still is increased, and the effluent liquor leaves at a higher temperature.

Given uniformity of seal throughout the chamber, or tray, 1 inch is certainly adequate. Absorption, as distinct from dissociation, arises with a too deep seal. Experiments indicate that the emergence of the steam-ammonia stream along the liquor line in the chamber, or trays, is calculated to afford ideal working conditions.

(2) *Wetted Surface Area*.—Obviously it is desirable to secure as large a surface area as possible within the limits of the size of the still, but it is doubtful whether wetted surface alone is the desideratum.

Fig. 44 shows a continuous gas liquor still which was in operation in London in 1878, the design of which was based on the principle of a comparatively large surface area with a thin film of liquor, along which the gases and steam skirted, by reason of the contiguity of the respective plates. It is known that a still 15 ft. high by 7 ft. by 5 ft. in section, having 38 plates, was capable of dealing with the free ammonia content of ten thousand gallons of gas liquor of 6 to 8 oz. strength per day.

It is clear from the experience afforded by the operation of the above still that heating surface is not alone the governing factor which should be aimed at in the design of gas liquor stills. Rather must attention be directed to ensuring the greatest intimacy of contact along with maximum time contact and heating surface. Attenuated bubbling of such a nature that maximum deformation and/or reformation of the bubbles are ensured is the principal factor for promoting rapid equilibrium.

(3) *Vapour Space above the Liquor in the Chambers*.—Clearly, there must be a well-defined vapour space above the liquor in the trays, in order to allow of equilibria being obtained, and of time contact between the two streams. It has been suggested that if intimacy of contact between the steam-ammonia stream and the liquor is to be secured the liquor surfaces should be extended, and the steam-ammonia spaces reduced. This is partly true, but the author's experiments still lead him to the view that a well-defined gas space is necessary, if only for the reason that it ensures time contact between the gas and liquor.

(4) *Vapour Velocity*.—The velocity of the steam-ammonia stream through the liquor in the trays influences the efficacy of the bubbling action. Optimum bubbling is a function of a definite vapour velocity. This velocity should not be such as will

cause entrainment, much less arrest the flow of the liquor, otherwise it would defeat its own object. Data having reference to vapour velocity will be given when discussing entrainment.

(5) *Entrainment*.—Entrainment may arise at the outlet of the still, or it may occur from tray to tray upwards. In either case it has adverse consequences. In the first instance cited, if entrainment is occurring in conjunction with a still which is attached to a sulphate of ammonia plant, it will be found difficult to avoid the formation of blue salt, as ferrocyanides and thiocyanates will be carried forward to the saturator, and as the acid feed to the latter invariably contains traces of iron, all the elements are present for the formation of ammonium ferrous-ferrocyanide, which, on exposure to the air, will lead to the formation of blue salt.

Some firms provide effective anti-priming devices in connection with their ammonia stills to obviate entrainment. Other makers prefer to adopt a void chamber at the top of the still, so as to reduce the velocity of the vapours, thereby largely effecting deposition of the mechanically entrained vapours.

Where entrainment takes place to any appreciable extent from tray to tray upwards the effect is twofold. Chas. Cooper, M.Sc., has stated the case very clearly in this connection (see *Proceedings of the Chemical Engineering Group*, vols. iii. and iv. pp. 106-107), and one cannot do better than quote his words. He says: "Where entrainment takes place in sensible quantity two effects are felt. One of these could best be appreciated by stating the limiting case, which is what would obtain if it were possible to blow the whole of the

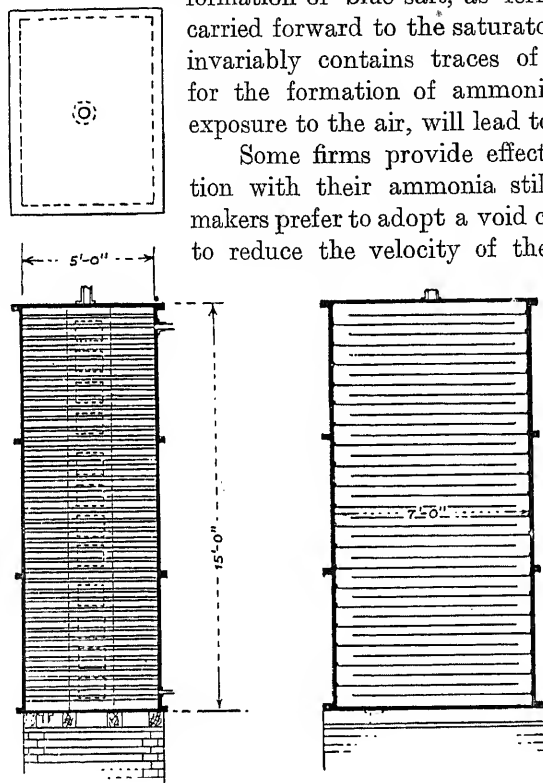


FIG. 44.—HILLS'S STILL.

liquor out of the still; a single stage action would be produced, and any given proportion of removal would be very wasteful in steam compared with a multiple stage treatment of the same gross capacity. In a more general form this proposition could be stated in this way: that in any counter-current process carried on in uninterrupted stages there is a downward limit of the number of stages which must be provided to produce a definite proportionate stripping along with any desired richness of product (in this case a steam-ammonia mixture).

"The first effect of entrainment is to blur the sharpness of the 'cut' between the stages, and the practical result is the same as if the gross capacity provided had been divided into a less number of sections.

"The second effect is one of dilution of the liquid in any section by the addition of weaker material from below, therefore producing the result that a greater quantity of liquor of lower strength is actually being stripped. These two considerations dictate at some point a limit on the amount of turbulence desirable in such a column."

The following table gives the amount of ammonia dissociated in the passage of liquor through the trays of a still operated by the author several years ago, when good working was secured :

TABLE 24

Tray No.	Free Ammonia.	Fixed Ammonia.
	Per cent.	Per cent.
1	0.88	0.26
2	0.79	0.26
3	0.67	0.26
4	0.60	0.26
All CO ₂ expelled at this stage.		
5	0.45	0.26
6	0.46	0.26

Before passing into the still, the liquor contained 1.20 per cent of free ammonia, 0.26 per cent of fixed ammonia. After leaving tray No. 6 the liquor entered the liming chamber, to which was fed a constant supply of milk of lime. This was thoroughly agitated with the liquor by means of live steam. The amount of ammonia dissociated in the fixed still is represented by the following figures :

TABLE 25

Tray No.	Per cent.	Tray No.	Per cent.
1	0.28	5	0.07
2	0.17	6	0.05
3	0.13	7	0.03
4	0.11		

It is seen that the above results represent satisfactory working, and that no serious entrainment from tray to tray is occurring. It is certain that with a good design of still the necessary turbulence can be secured without causing entrainment.

Experimental Determinations of (A) Vapour Velocity causing Incipient Entrainment, and (B) the Volume of such Entrainment at varying Gas Velocities

(A) *Experiment.*—For these determinations, a section of an experimental still (see fig. 45) was provided. The section consisted of three trays with gas bubblers arranged transversely, with liquor inlets and overflows, and with an air inlet at H.

The area of the serrations in each bubbling hood was measured so as to give a basis for the expression "gas velocity." Each hood had triangular serrations $\frac{3}{8}$ in. by $\frac{3}{4}$ in., the total area being 3.2 sq. in.

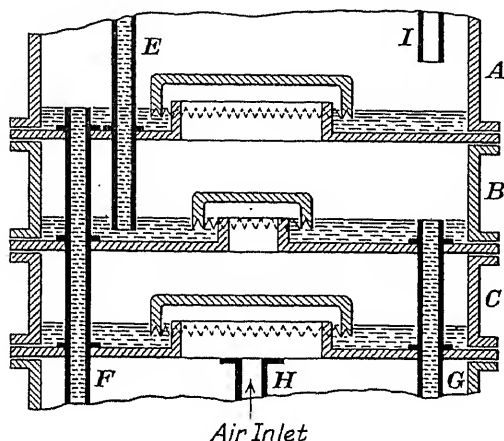


FIG. 45.—SECTION OF EXPERIMENTAL STILL.

A preliminary experiment was made with dilute iodine solution, using starch as an indicator. The tray B was filled with iodine solution to the overflow, while A was similarly filled with starch solution.

Air was passed in at H for 15-minute periods at increasing velocities until coloration of the starch solution showed that entrainment was taking place.

The following is a list of the velocities so obtained, the last of the series being that at which incipient entrainment commenced.

- | | | | | | |
|-----|------|----------|---|-------|-------------------------|
| (1) | 0.83 | cub. ft. | = | 37.6 | linear feet per minute. |
| (2) | 1.32 | " | = | 59.7 | " " " |
| (3) | 1.87 | " | = | 84.8 | " " " |
| (4) | 2.30 | " | = | 104.2 | " " " |

This experiment was repeated with AgNO_3 and NaCl solutions in place of iodine and starch, (1) to prove that volatilisation of the iodine was not the cause of coloration, and (2) to find values intermediate between 84.8 and 104.2.

Three velocities were tried, commencing with 79 linear feet per minute.

- | | | | | | |
|-----|------|----------|---|-------|-------------------------|
| (1) | 1.75 | cub. ft. | = | 79.2 | linear feet per minute. |
| (2) | 2.03 | " | = | 93.2 | " " " |
| (3) | 2.17 | " | = | 118.8 | " " " |

At 118.8 linear feet per minute entrainment was evidenced by the precipitation of AgCl in the section A.

(B) *Experiment*.—In order to determine the volume of liquor carried upward from tray to tray by reason of entrainment, two measured solutions were circulated in the apparatus used in the previous experiment, and after blowing air at known velocities for definite periods, increase in the volume of one solution and corresponding decrease in the volume of the other was noted.

Water was circulated through A by means of the inlet I and overflow F, and through B by the inlet at E and overflow at G.

The rate of water circulation was approximately 6 litres per hour, and air velocity 120 linear feet per minute.

The experiment was carried out for two hours, at the end of which time there was no appreciable reduction in the volume circulated in B.

A further two hours' period was therefore run, with the air velocity increased to 140 linear feet per minute.

Both A and B volumes were then measured, and it was found that B had lost 75 c.c., while A had increased 62 c.c. It was assumed that the difference (13 c.c.) had been lost through evaporation, and 75 c.c. was taken as the extent of the entrainment.

75 c.c. in 2 hours = 37.50 c.c. per hour.

37.5 c.c. on 6 litres = 0.63 per cent.

It is clear from the above experiments that incipient entrainment occurs at about 2 linear feet per second, but the entrainment is negligible, and it is not improbable that much higher velocities can be attained before the entrainment need cause any concern from the point of view of steam consumption.

From calculations which have been made of various designs of ammonia stills, it would appear that these allow of maximum gas velocities of from 2 to 30, with minimum gas velocities of from 1 to 15. It would appear that a figure of about 10 ft. per second would represent best practice for maximum velocity, and about 5 ft. per second for minimum velocity.

(6) *Time Contact between the Steam-Ammonia Stream and the Liquor on the Trays.*—This is an important factor in aiding the attainment of equilibria between the two systems.

This concerns, and is a complement of, consideration No. 3.

(7) *Form of Bubbling Device.*—Much depends on the form of distribution device adopted, as to whether sluggish or rapid equilibria are attained. In the past it has been usual to adopt either rectangular slots or serrations. The sensitiveness of a serrated hood does not appear to have been adequately appreciated. Practical observations have revealed that with a slope, or if the hood is partially displaced, or if there is some minor fault in level, causing a tilting of the distributing device, the steam-ammonia stream will be discharged on the narrow portion in the form of relatively large bubbles. Under such conditions there is a good deal of entrainment.

If, on the other hand, the size of the bubbles by a suitable distributing device is reduced to a minimum, equilibria are established in a minimum of time. Various types of "frogs," etc. have been tried from time to time, and it has been found that a distributing device with holes of small diameter arranged as shown in fig. 46 not only gives an extremely fine state of division, but admits of impact between the respective bubbles, and generally aids much more rapid equilibria.

This kind of bubbler is satisfactory for the free section of an ammonia still, but it is unsuitable for the fixed portion, on account of its liability to blockage. An almost identical effect can be obtained by cutting slots about $1\frac{1}{4}$ in. deep by $\frac{3}{8}$ in. wide.

(8) *Single v. Multiple Gas Passages.*—Important as the above consideration is, it can be nullified to a large extent if the gas passages are not satisfactorily proportioned to give the desired vapour velocity. This raises the question of single as contrasted with multiple gas passages. Almost invariably, where multiple gas passages are

provided, the velocity of the vapour through the surfaces of the liquor is slow. This may be desirable from the point of view of preventing entrainment from tray to tray upwards, but experiments which the author has conducted indicate that short circuiting is very liable, and as one of the potent causes of high steam consumption is attributable to short circuiting, this is a factor which cannot be ignored.

(9) *Definite Capacity*.—Obviously there is a definite capacity for each particular still, above which and below which its operation becomes inefficient. What that capacity is should be definitely determined by the makers, and all users should be clearly advised of the consequences following a departure from the recognised capacity.

It will be found that steam consumption is a function of the definite capacity of the still. Working under a specific capacity sensibly accentuates short circuiting, besides which an increased loss through radiation occurs. Operating above the specific capacity invariably results in loss of ammonia through the waste liquor.

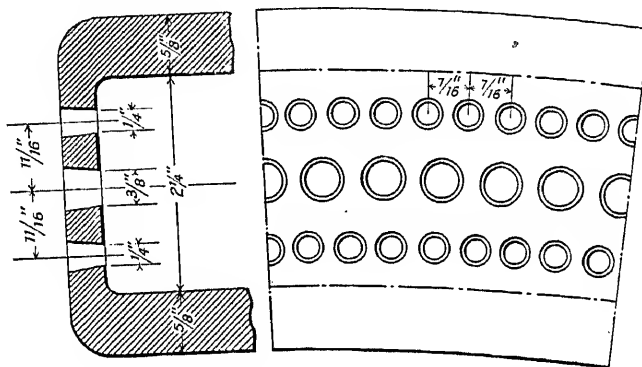


FIG. 46.—DETAILS OF HOLES IN BUBBLER (DR. CARPENTER'S DESIGN).

From inquiries which the author has instituted it is feared that the question of definite capacity has not received the attention which its importance demands; and it is suggested that investigation of this phase of still design would be prolific of good results.

(10) *Size of Stills in Relation to Capacity*.—It is not proposed to enter into the details of this aspect, except to give particulars of the practice followed by several makers of stills. Examination of the various particulars suggests that in this respect also there is need for investigation. To increase the capacity of a still, the height invariably remains unaltered, but the diameter is varied. Details of sizes are given below :

TABLE 26

FIRM "A"

6 ft. diam. still deals with 90 tons of gas liquor of 2.13 per cent NH_3 per day.

5	"	"	"	70	"	"	"	"
4	"	"	"	50	"	"	"	"

TABLE 27

FIRM "B"

3 ft. 6 in. diam. still deals with 50 tons of gas liquor of 1.73 per cent NH_3 per day.
 3 ft. " " " 33 " " " " "

TABLE 28

FIRM "C"

1 ft. 6 in. diam. still—6 tons per day maximum capacity.
 2 ft. " 12 " " "
 3 ft. " 24 " " "

This firm also makes square stills as follows :

3 ft. square—30 tons per day maximum capacity.
 4 " 50 " " "
 5 ft. 6 in. square—75 tons per day maximum capacity.

TABLE 29

FIRM "D"

4 ft. diam. still for dealing with 30 tons of 2.1 per cent liquor per day.

For dealing with 50, 70, and 90 tons per day, the square type of still is recommended. The size is always 5 ft. 6 in. square, and the height is varied according to the size required.

TABLE 30

FIRM "E"

Tons per day.	Galls.	Size.	Area. Sq. ft.	Cub. ft.	Area. Sq. ft. per ton.	Cub. ft. per ton.
80	17,600	5 ft. diam. \times 19 ft.	19	361	.24	5.9
60	13,200	5½ ft. sq. \times 12½ ft.	30	375	.50	6.2
80	17,600	5¾ ft. diam. \times 20 ft.	26	520	.32	6.5
50	11,000	4 ft. diam. \times 18 ft.	12½	225	.25	4.5
50	11,000	4 ft. diam. \times 14½ ft.	12½	182	.25	3.6
50	11,000	4 ft. sq. \times 15 ft.	16	240	.32	4.8
56	12,200	4 ft. diam. \times 16 ft.	12½	200	.222	3.57

(11) *Bubbling v. Surface Area via Packed Columns.*—The author has had many opportunities of studying the operation of ammonia stills, and other such apparatus, where the principle of bubbling was on trial as compared with that of surface area via a packed column. While it is true that many stills constituted of a packed column can be made to operate satisfactorily, the tendency to channelling with such stills, apart from the larger size necessary for the dissociation of a unit weight of ammonia, is a distinct disadvantage. For a given size, there is no doubt that by adopting the principle of bubbling a sensible increase of capacity is secured.

The principle of a packed column for ammonia distillation is exemplified in B.P. 8317, 1907. In this instance Wyld and Shepherd provided for a still (fig. 47) comprising a number of secure cast-iron sections forming the shell. The still, contrary to usual practice, is not divided

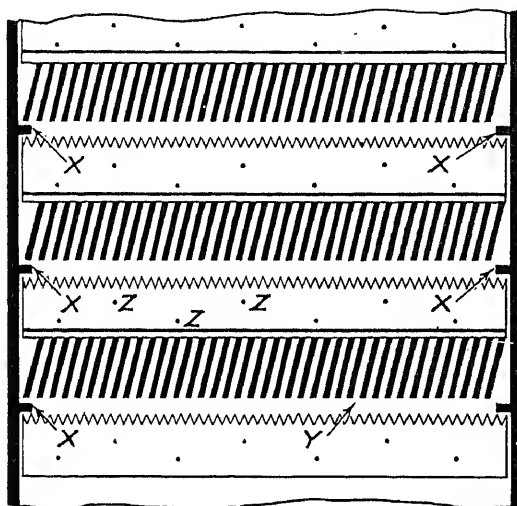


FIG. 47.—WYLD AND SHEPHERD'S STILL.

into a number of chambers, but is furnished internally with a series of cast-iron ledges—X—placed alternately on the corresponding sides at predetermined centres. On these ledges rest thin cast-iron plates Y, the distance between which is governed by a raised surface at various centres Z. One row is placed transversely to the other. The plates are set on the incline at an angle of about 75°. With efficient distribution of the liquor, it will be appreciated that a very thin film, or wetted surface, is secured, and the contact between the gas and the liquor is large. Although this still has many features of interest,

it does not appear to have been largely adopted.

It will be seen from the foregoing that there are a variety of considerations to which attention must be directed in the design of an ammonia still. It is believed that nothing approaching finality in still design will be possible, pending some attempt to express the various factors either arithmetically or mathematically. Even so, judicious correlation will be necessary, and this must be regarded largely from the point of view of steam consumption.

AMMONIA STILLS

It will now be opportune to examine the various types of ammonia stills which are placed upon the market. If such examination is pursued in something like chronological order, it may be possible to determine whether there has been any appreciable and systematic evolution in this connection, or whether, as has been

suggested earlier in this chapter, the broad result has been confusion rather than simplification of types.

Savalle's column, introduced in 1875, has already been referred to. Following

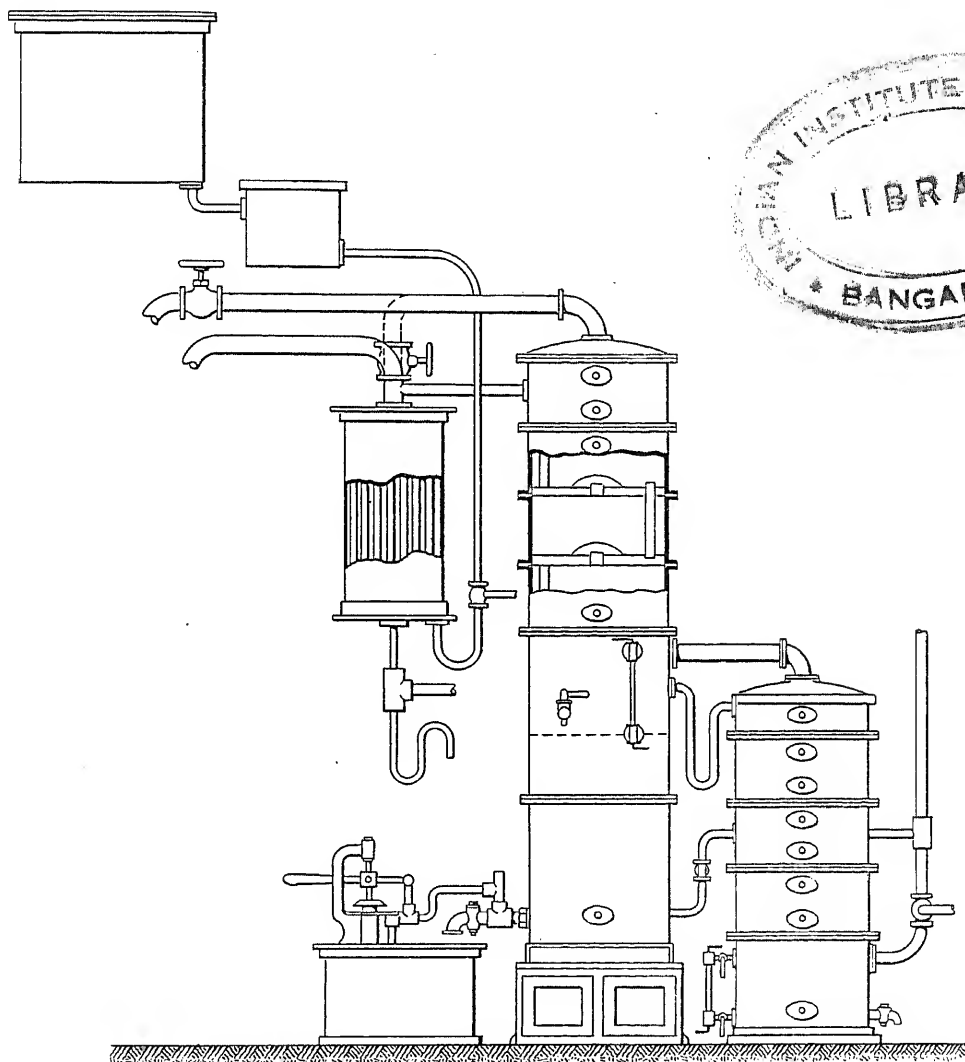


FIG. 48.—FELDMAN'S AMMONIA STILL.

this, patents were granted to Clark (1876), and Young (1881 and 1883), but these respectively have reference to the arrangement of a series of stills in connection with the distillation of sewage liquors, and to the partial desulphurisation of gas liquor. In 1882 the Feldman still (B.P. 3643) was introduced to this country.

This apparatus embodies the general principles upon which most of the continuous stills are based. Fig. 48 illustrates the still in question. It will be seen that the apparatus comes under type "A." The gas liquor to be distilled passes from tray to tray by means of a single flow, and the steam-ammonia stream ascends by a single gas passage. In both these respects the provisions appear to be commendable.

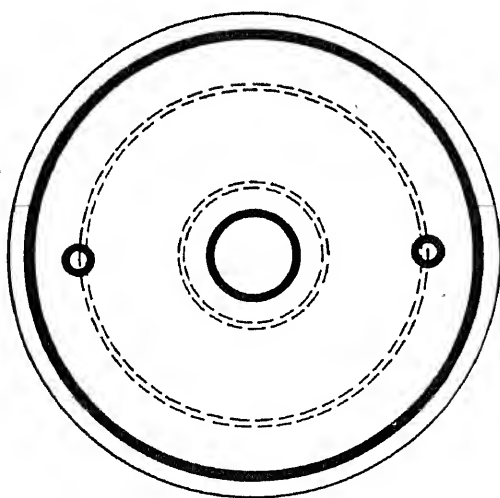
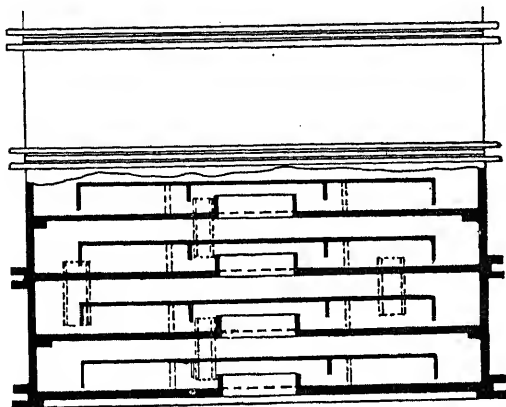


FIG. 49.—ELEMENTS OF GAS LIQUOR STILL.

The liming chamber, according to the patent specification, is similar to the one shown in fig. 105. To introduce intermingling of the liquor with the milk of lime, and to promote decomposition of the former with the latter, a steam recoil wheel is provided in the lower part of the liming chamber. The action of the recoil wheel is based on the principle of Segner's water wheel, or Barker's mill. A sieved bottom is provided about two-thirds the height of the liming chamber, so as to impede the movements of the liquor in this part, thus enabling the agitated milk of lime to settle more rapidly in the upper part of the chamber.

The limed liquor overflows through a sealed pipe into the fixed still and passes from tray to tray. Steam is introduced to the bottom of the chamber and ascends through single gas passages in the same way as is the case with the free still.

The Grünberg still was also introduced in this country about this time. The characteristics of this still are included in Table 19, being the first series of figures under category "B." Fig. 49 gives details of the elements of the free column.

In 1887 Davis (a former Chief Alkali Inspector) was granted a patent, No. 15540, for an ammonia still. This is illustrated in fig. 50. Davis's invention was directed to securing a much finer state of division of the ammoniacal vapours in order to get the utmost possible work out of a given size of still. In the first portion of his complete specifications Davis says: "In the stills for the evolution of ammonia gas, or of carbonate or sulphide of ammonium, from any of their solutions, it has been

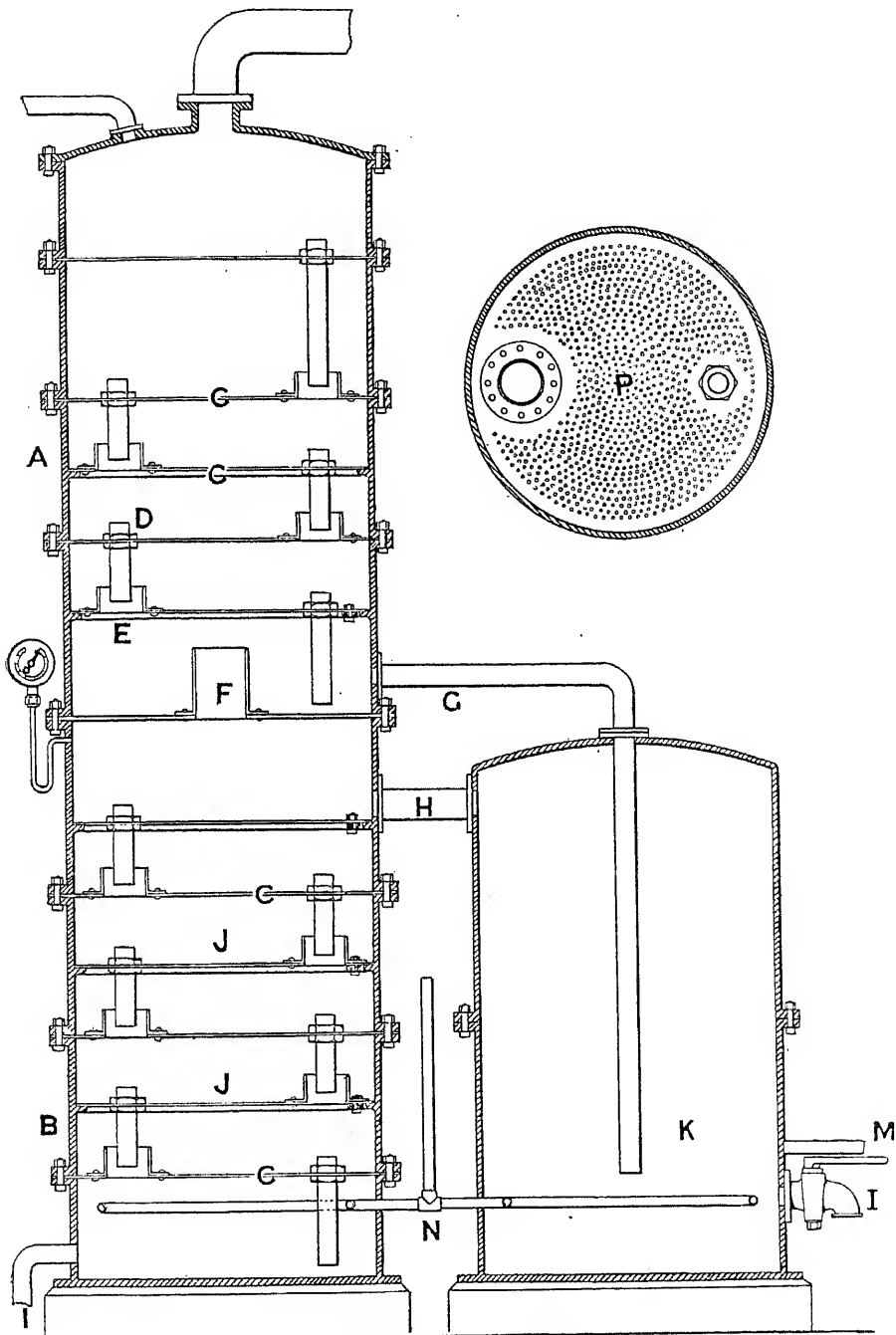


FIG. 50.—DAVIS AMMONIA STILL.

the custom either to drive off the volatile substances by means of a jet of steam blowing direct on to the liquid, contained in a boiler of the usual pattern, or to heat the liquid by means of a direct fire, or to pass the steam and vapours up a tower or column filled with bricks or boards, down which the ammoniacal liquid is made to pass, or to cause the liquid to run down a column containing shelves pierced with large central apertures, covered with caps, serrated round their lower edges, through which the vapours are made to pass."

Davis provided for the construction of a still by which a much larger quantity of ammoniacal liquor could be distilled in a given space than heretofore, and with ordinary care could never become choked with tar. It will be seen from fig. 51 that

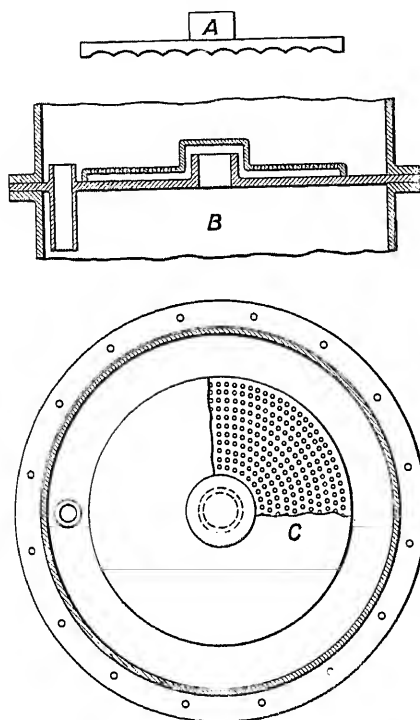


FIG. 51.—DAVIS STILL (PERFORATED PLATES).

which allow of the escape of the liquor but retain the steam.

The essence of this invention is the use of a column still divided in two parts in combination with a vessel for lime. The still is provided either with perforated trays or perforated caps on which the liquor, both before and after mixing with lime or caustic soda, is boiled by means of steam in the chamber immediately below it. The application of a series of one or more U-tubes to the outlet of the still is also covered.

Two points of importance occur to the author in connection with the design of this still, particularly when perforated trays are used. In the first place, experience indicates that in the actual working of a perforated tray still difficulties occur. If

this still comes under category "C." A represents the free still, B the fixed still, and K the outside liming chamber. The stills are provided with a number of trays C. Each tray is fitted with an overflow pipe D, dipping into a shallow well E, and these trays are perforated with a number of small holes, as shown in the plan P, through which the vapours pass when the still is at work. Davis found that about one hole, $\frac{1}{16}$ in. diameter, in every square inch of tray gives the best results, but an important omission would appear to be the quantity of liquor distilled in unit time per square foot of tray so perforated.

The volatile ammonia is driven off in the top portion of still A, and the partly freed liquor passing through the pipe C into the lime vessel becomes mixed with lime introduced through the pipe M. From K the liquor re-enters the column by pipe H, and in descending the still, over the perforated trays, becomes denuded of its fixed ammonia, which is discharged from the column at the outlet I by means of a series of U-tubes

the still is operated at a quicker rate than that for which the holes are designed, the chambers begin to accumulate liquor, and incidentally extra steam pressure is required.

On the other hand, if the operation is too slow, the liquor throughout the whole column drains away, leaving nothing on the plates. It is seen that the flexibility of this type of still is very small. Apart from this disability, in the fixed portion of the still it is not improbable that trouble arises owing to blockage of the perforations.

Davis was evidently aware of these difficulties, because it is found that to overcome possible irregularity of feed of the liquor he provided for the use of a perforated cap B, but even this device lent itself to easy blockage in the fixed portion of the still.

Blockage trouble due to imperfect decarbonation and to limed liquor for the fixed still were the subject of consideration in the early years of the development of the continuous column still. It is known that Mallet, in 1884, devised a "colonne inobstruable," which would not only serve to avoid the difficulties indicated, but would allow of the initial liming of the ammoniacal liquor. This latter operation is necessary with sewage liquors in order to admit of the precipitation of phosphates and other such bodies. The advantage of the Mallet "colonne agitée," illustrated in fig. 52, is that there is no necessity to clarify by repose, nor indeed is it essential to relegate the lime treatment to another apparatus. The ordinary column *b, b*, on which rests an analyser *a*, allows of the expulsion of the volatile ammonia. The liquor from the latter vessel passes by *c* into an apparatus intended for admixture with lime. The vessel is conical in character, and is charged with such a quantity of calcium oxide as to serve for several hours. An endless screw affords a positive feed to the mixer. The liquid enters through the sealed pipe, slaking the lime which is carried in the form of cream of lime to the rotary shaft column. By operation of the cock at the foot of the lime agitator it is possible to remove unslaked lime, stones, and ashes, etc. The limed liquor now descends the lower column, and the rotary shaft with rabbles keeps the lime in suspension in the liquor, and obviates the formation of deposit or laminations on the plates. The effluent liquor is suitably discharged. With very dirty liquors a sludge box is indispensable, from which sludge can be discharged from time to time.

While many chemical manufacturers may not be enamoured of a mechanically operated column, without doubt the latter has many advantages, and it is known in England that one or two works speak in commendatory terms of the Mallet column agitator.

It should be remarked that there is a distinct advantage in introducing the lime direct to a vessel which feeds the distilling column. This aspect has been enlarged upon in Chapter VIII.

In 1887 Walker was successful in securing a patent, No. 16806, for a still in which the employment of hoods carried by a vertical shaft was provided for. This still is shown in fig. 53, and it is seen that in addition to the above feature it embraces the use of injectors for the agitation of the liming section of the plant.

It is necessary now to direct attention to the Wilton's still, for which a patent (No. 24832) was granted in 1901. Figs. 54 and 33 show clearly the features of this still. The hood is of elongated form, placed in the centre of the tray, and the inlet

and outlet orifices are arranged at opposite sides of the chambers. Ample provision is made for the removal of the hood, as indeed for clearing any obstruction of the overflow pipes.

One disability appears to be that the area of the serrations is equal to, if not in excess of, the area of the gas hoods. Thus the vapour velocity is inclined to be somewhat sluggish.

W. C. Holmes & Co., Ltd., supply a variety of stills for specific purposes. Fig. 55 is suitable for a small works. The various sections in both stills are held together by vertical tie-bolts. Stills of this type should not be screwed up tight when cold, but allowance should be made for expansion. In the event of slight leakages the nuts on the tie-bolts can be screwed up with a spanner. Invariably springs are placed under the nuts, in order to afford sufficient latitude in the matter of expansion and contraction. Another circular still made by the same makers is shown in fig. 56, which represents a 5-ft. diameter still with fairly large manholes. Fig. 57 represents a square type of still, each chamber of which is provided with four bubbling hoods, and the liquor traverses the trays in a zigzag fashion. Fig. 58 shows the connection of the latter type of still to an outside lining chamber. Such an arrangement comes under category "C."

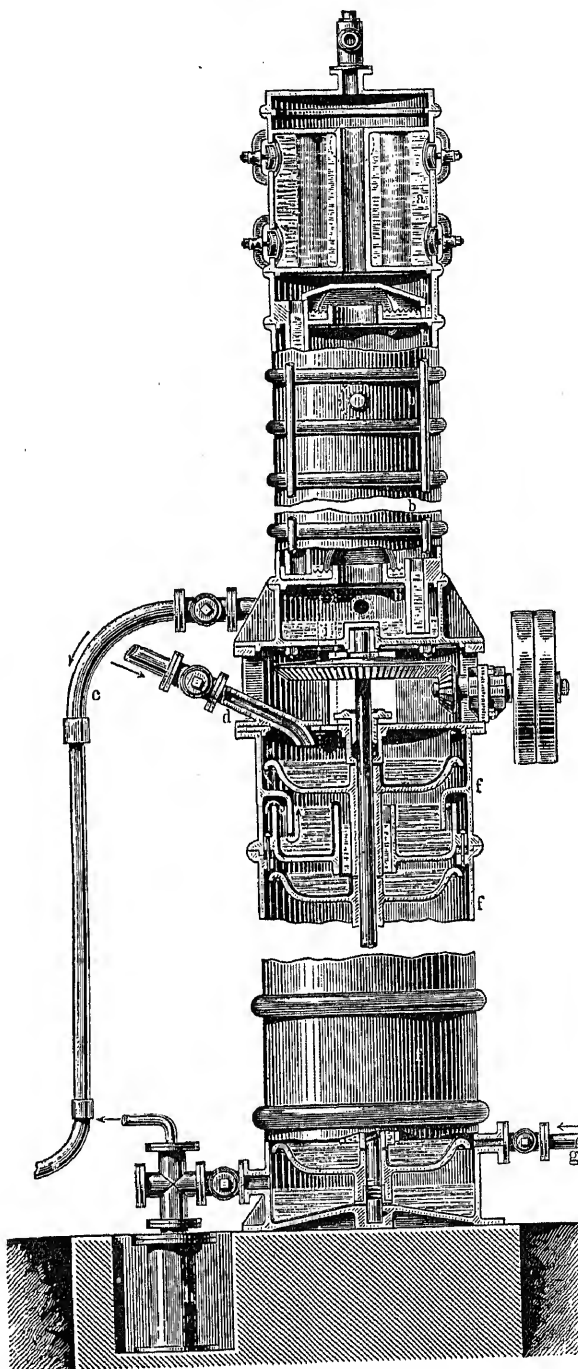


FIG. 52.—MALLET "COLONNE INOBTURABLE" FOR AMMONIA DISTILLATION.

To traverse the various features of all stills is hardly possible. Those of Colson (made by Ashmore, Benson & Pease), Dempster, Walker, and Koppers (B.P. 2399, 1908) have much in common. Some of these will be dealt with under the subsequent heading.

ELEMENTS OF AMMONIA STILLS

Fig. 59 shows how the elements of the still illustrated in fig. 36 are assembled. The bottom tray is supported on a circular webbed flange arranged within the still. Each succeeding tray is in turn supported by the one underneath. The first bubbler of the free still, placed immediately above the liming chamber, is supported by a vertical 9-in. cast-iron pipe (which, by a bend near the foot, conducts the limed liquor to an outer annulus) placed centrally in the liming chamber and terminating with a suitable base. The first bubbler supports the hood, which in turn supports the next bubbler. Thus the whole weight of the bubblers and hoods is transmitted to the 9-in. pipe, and the complete weight of the trays is reflected on the webbed flange. For the permanent machined joints a mixture of red and white lead is used. For the faced joints of the superimposed sections graphite paste is used. A packing of yarn is also employed between the trays and the outside shell of the still, so as to prevent the admission of any corrosion which will interfere with the removal of the trays. It should be remarked that the bubblers of the fixed section of this particular still are open, with serrated edges on the two peripheries.

The merits of the design of this still are the size of the liquor overflows and the

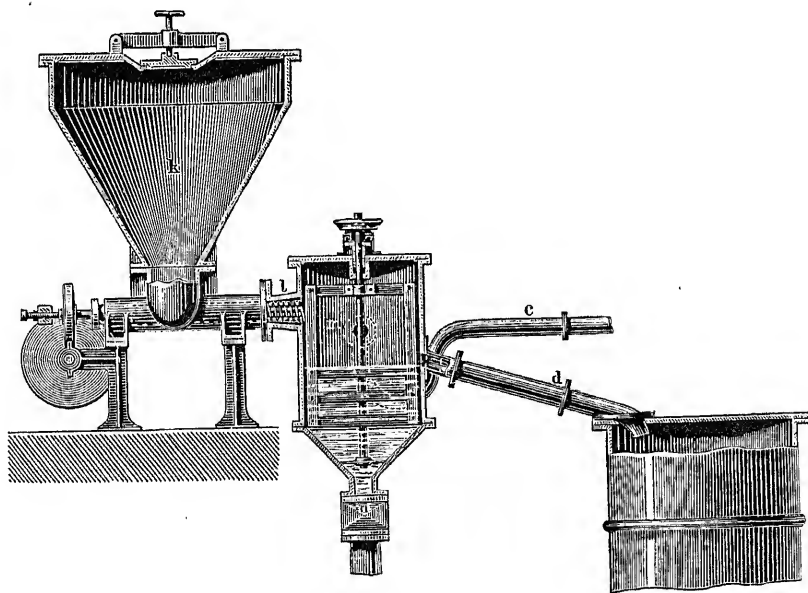


FIG. 52.—MALLET "COLONNE INOBSURABLE" FOR AMMONIA DISTILLATION.

large area of wetted surface which the elements afford. Further, it is evident that the designer has appreciated two other important factors, viz. the great advantage of a specific velocity of the vapours where they come in contact with the liquid, and the importance of time contact as between the vapour above the liquid and the liquid itself as aiding equilibrium.

Fig. 49 shows the elements of a still which was introduced to this country about forty years ago. Some of the features of this still, particularly the liming section,

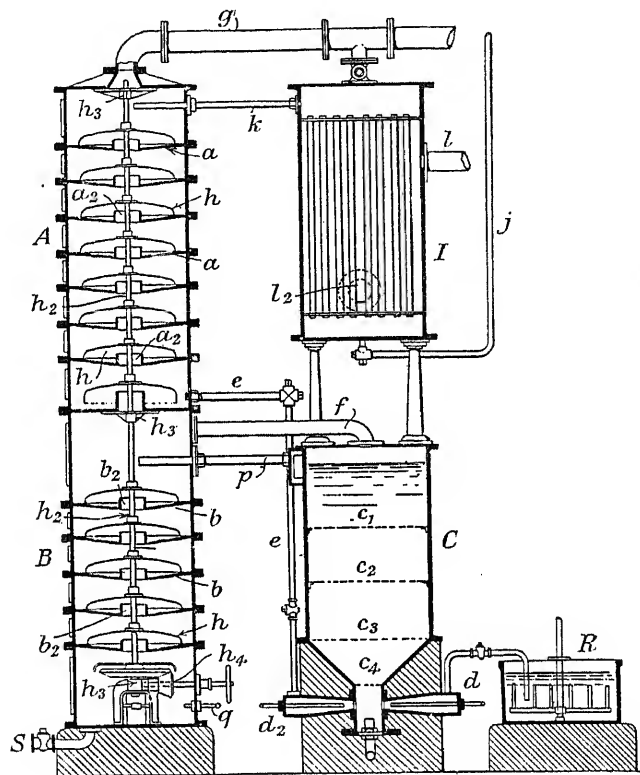


FIG. 53.—WALKER'S ROTARY STILL.

possess definite merit, but it is believed that there are many directions in which the still can be improved, particularly in the matter of area of liquor overflows, time contact between the vapour and liquid, and velocity of the vapours through the liquid.

Fig. 60 represents the elements of a still of more recent design. Regard has obviously been paid to the removal of the hoods for cleaning purposes, and the possibility of blocked outlets would appear to be remote, in view of their size. The comparatively small diameter of the still in relation to the quantity of gas liquor with which it deals, and the arrangement of hoods which clearly admit of the bubbles

from one being thrown in contact with those created by the adjacent hood, must ensure considerable deformation, thus increasing the contact of liquid and vapour.

Fig. 61 represents a very simple form of element. If simplicity were the pre-

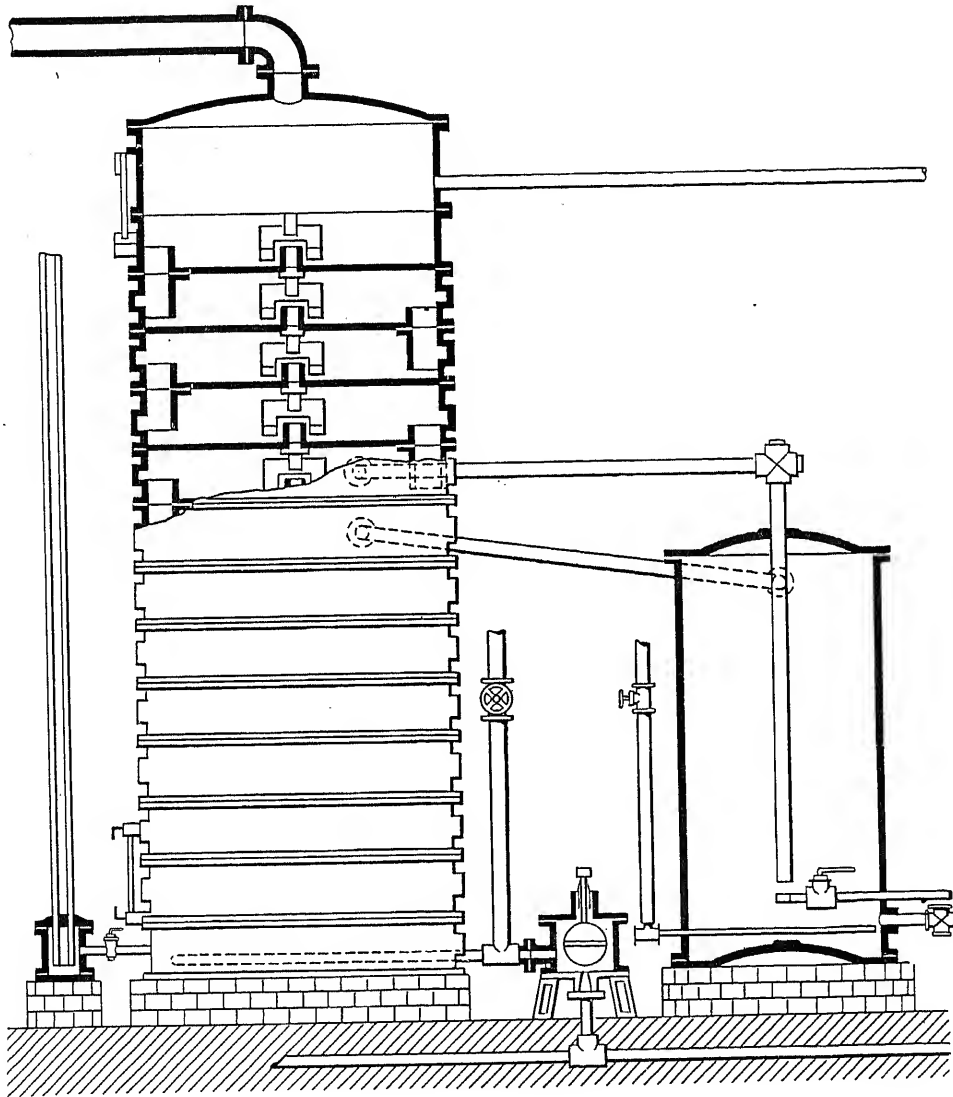


FIG. 54.—WILTON'S STILL.

dominant factor in the efficiency of a gas liquor still, this particular design would certainly merit a premier place. The hoods in both the free and fixed stills are rectangular and the travel of the liquid is left to right and right to left alternately. The

overflows are tolerably large, and the designer has evidently had in mind the import-

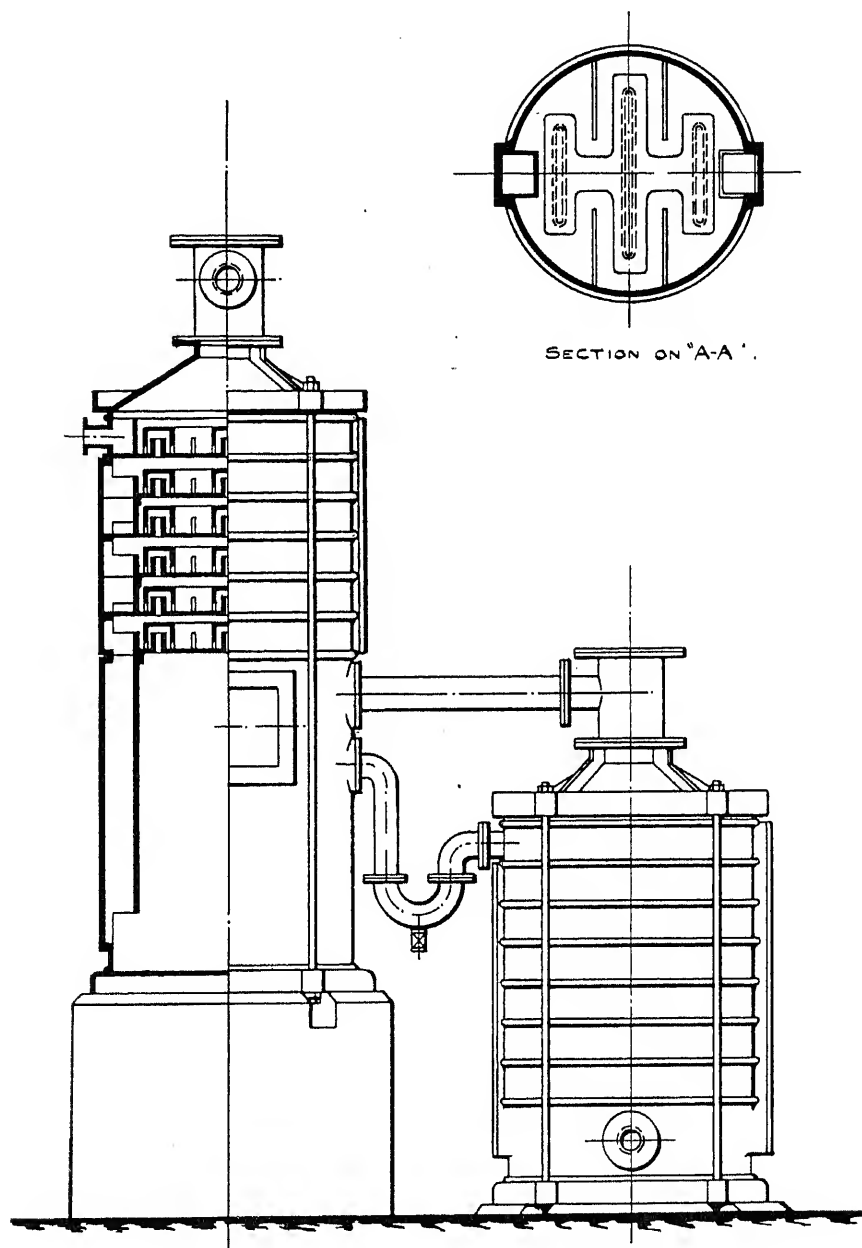


FIG. 55.—HOLMES'S STILL FOR SMALL WORKS.

ance of avoiding blockages at this point. The hoods are capable of ready removal.

One disadvantage appears to be that the arrangement of hoods lends itself to localised bubbling. Moreover, the area of the serrations is equal to, if not in excess of, the

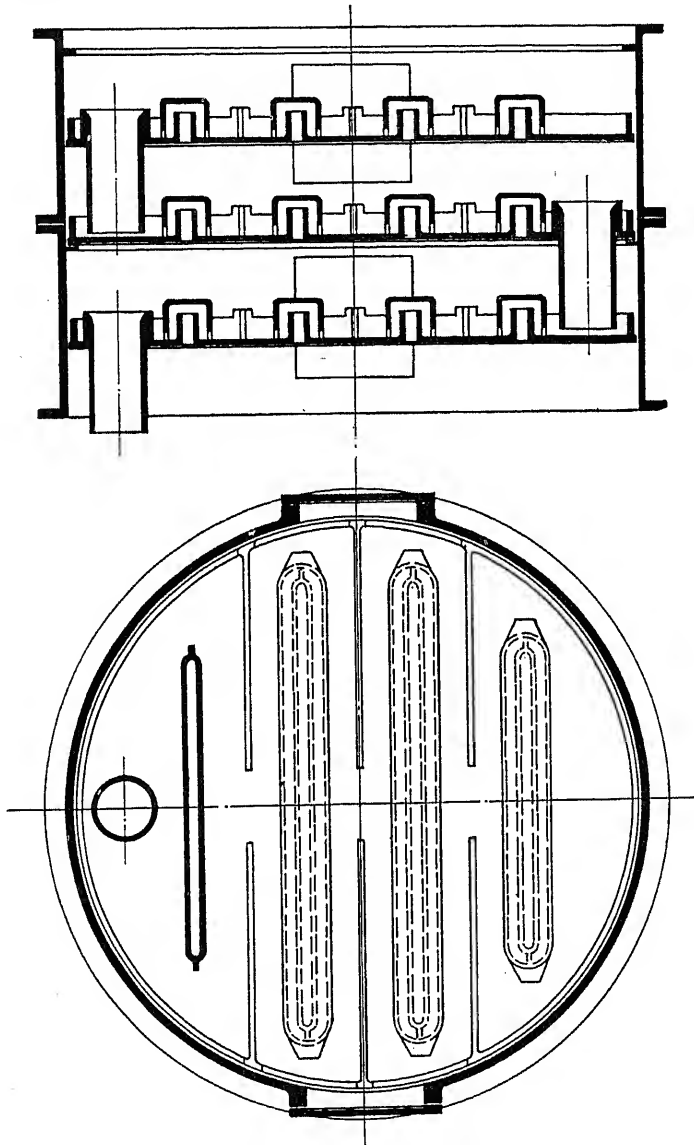


FIG. 56.—HOLMES'S 5' 0" DIAMETER STILL.

area of the gas slots, which area is too large for ensuring such a vapour velocity as to give optimum bubbling.

Fig. 62 represents the elements of a square still which, by reason of its form, must have the merit of cheapness as regards cost of production. The bubbling

hoods are also readily accessible, either for inspection or withdrawal. Whether the liquid will follow the path intended for it appears open to doubt. While the arrangement of four hoods is a considerable advantage, it is feared that the area for the ascent of the gas is far too large for ensuring satisfactory bubbling, and the

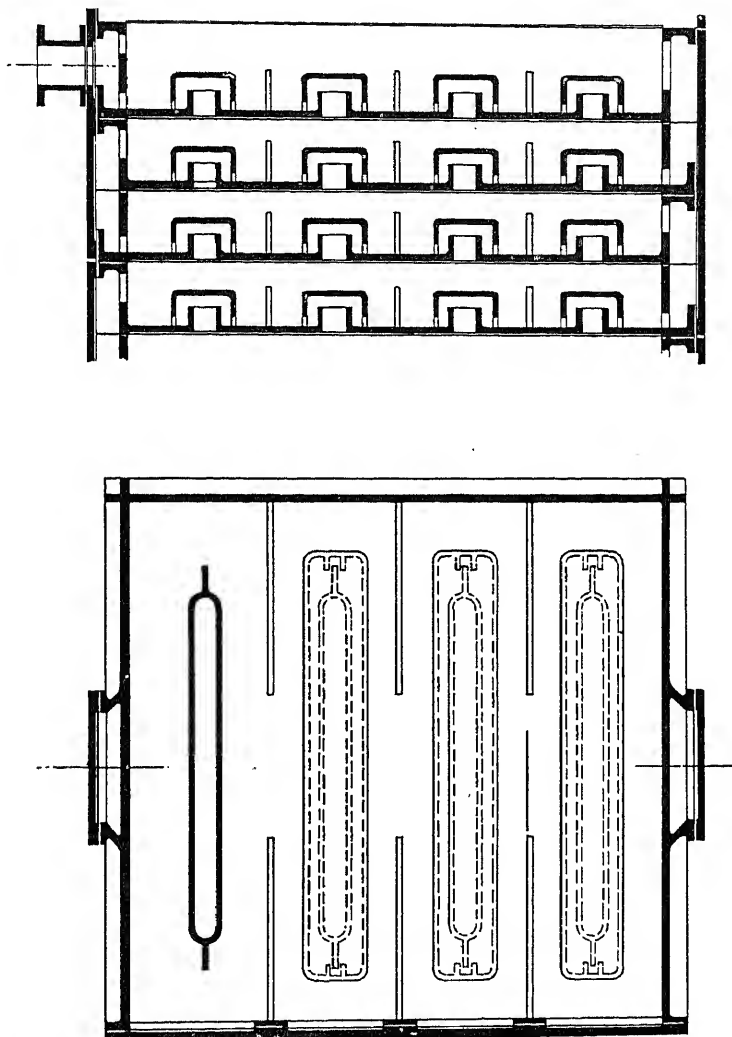


FIG. 57.—HOLMES'S SQUARE STILL WITH BUBBLING HOODS.

liquor overflows also appear to be unnecessarily restricted, although, protruding as they do beyond the castings, they are without doubt easy of access.

Fig. 63 represents the constituent parts of a still of recent design. This still has a very large capacity. The designers have recognised the importance of large

overflows and the advantage attending the depth of chamber in relation to the liquor

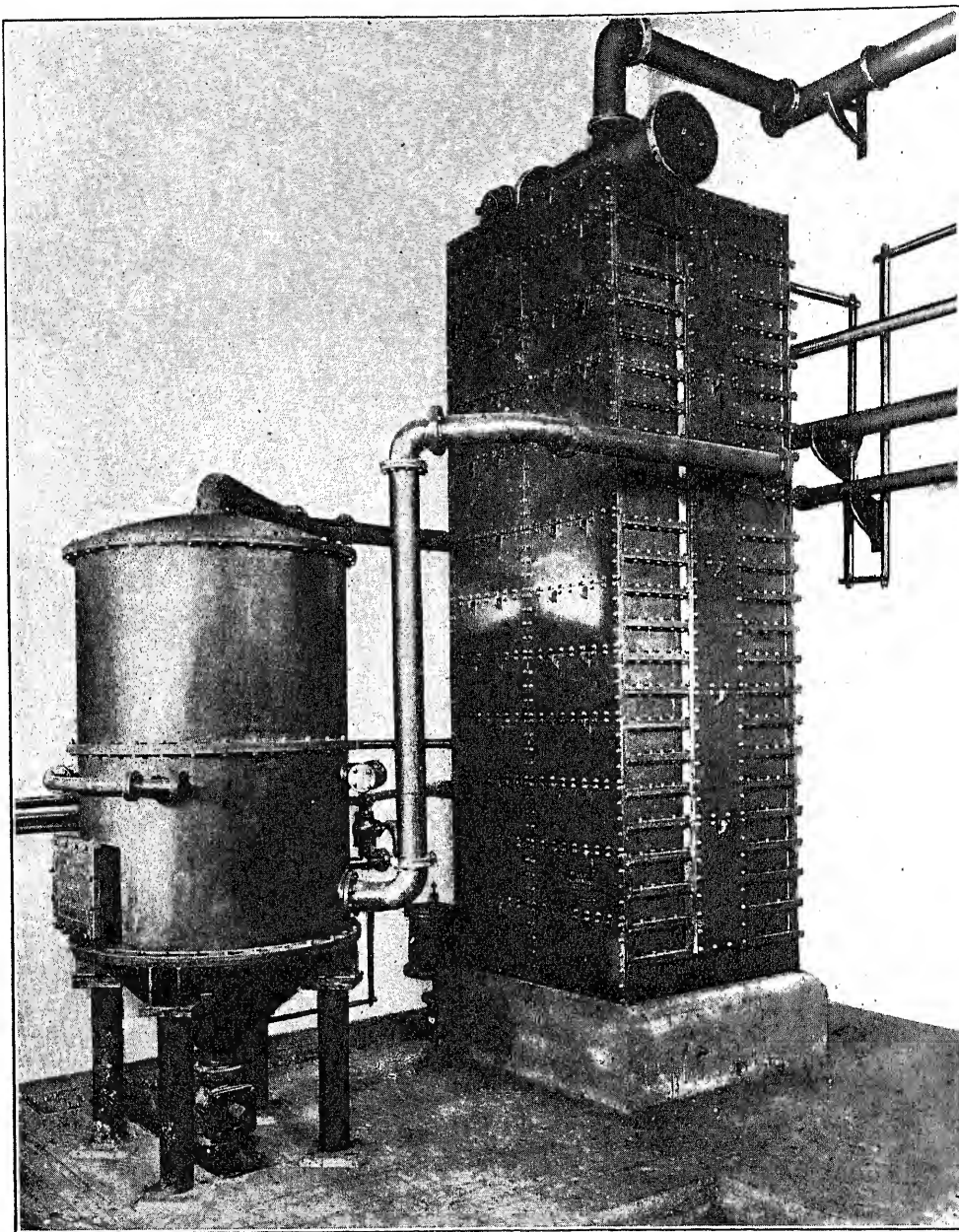


FIG. 58.—CONNECTION OF SQUARE STILL TO LIMING CHAMBER.

on the trays as increasing the factor of time contact. Moreover, the weight of the

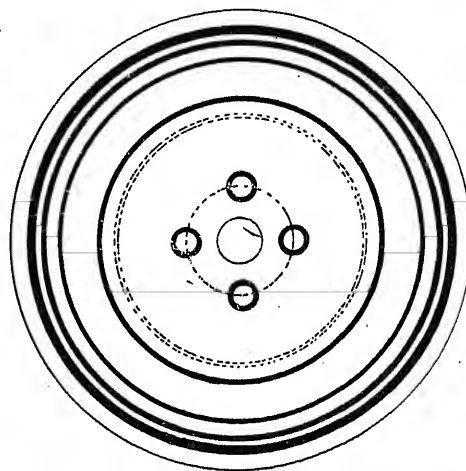
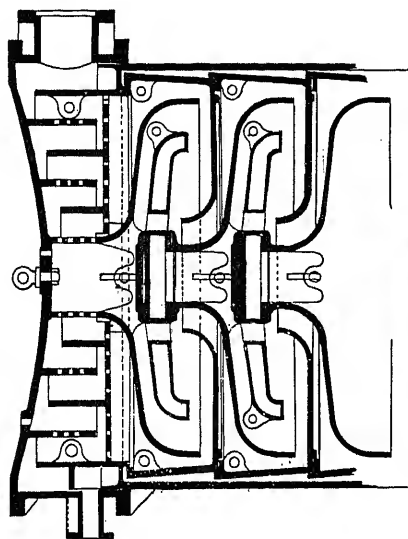


FIG. 59.—ELEMENTS OF GAS LIQUOR STILLS.

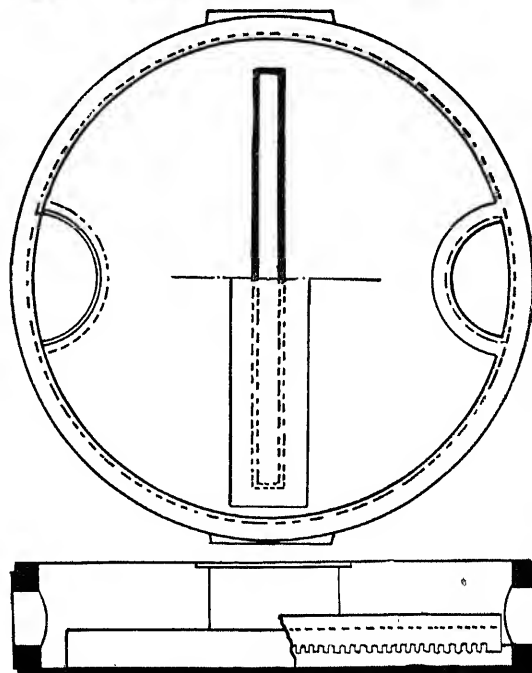
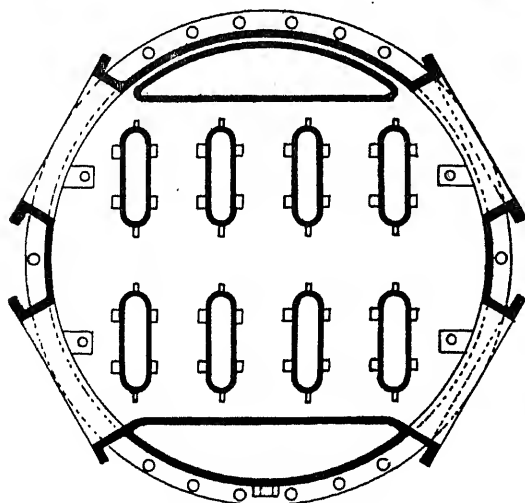
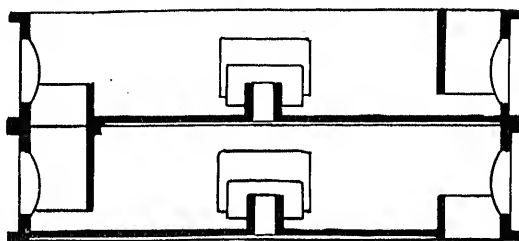
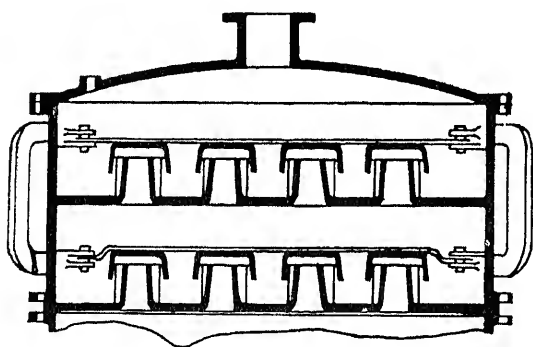


FIG. 60.—ELEMENTS OF GAS LIQUOR STILLS.

FIG. 61.—ELEMENTS OF GAS LIQUOR STILLS.

still in relation to the capacity is certainly a chemical engineering achievement.

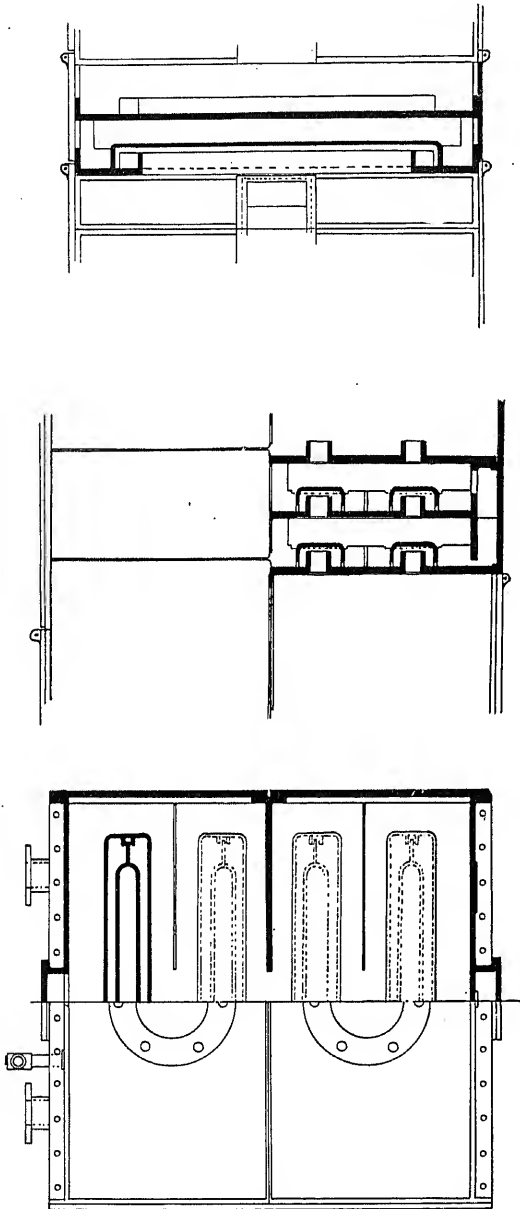


FIG. 62.—ELEMENTS OF GAS LIQUOR STILLs.

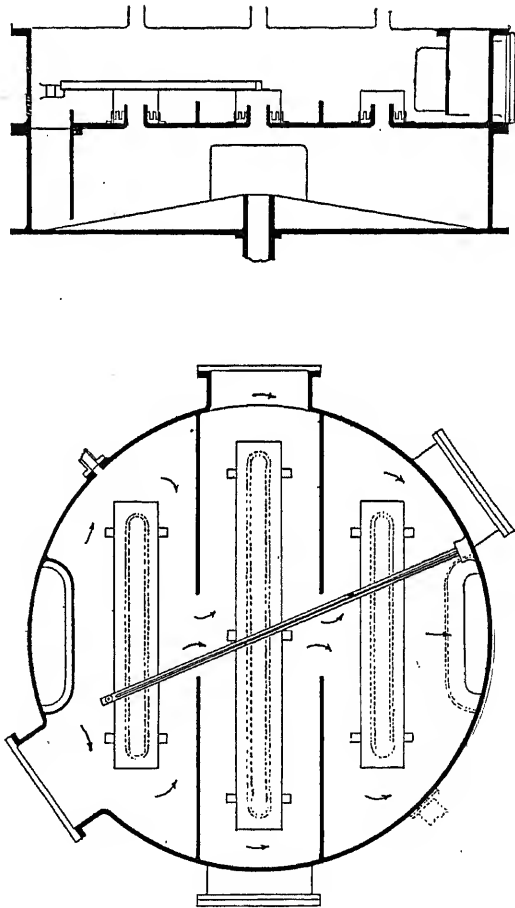


FIG. 63.—ELEMENTS OF GAS LIQUOR STILLs.

The latter is largely due to the adoption of a small liming chamber, and of course introduces a subject on which there would be, no doubt, considerable divergence of

views. It is believed that the design, by reason of the large area afforded by the serrations, militates against such a vapour velocity as is calculated to ensure satisfactory bubbling.

Fig. 64 represents the elements of a still possessing several merits, particularly the one of creating bubbling over a large surface area, and ensuring impact between

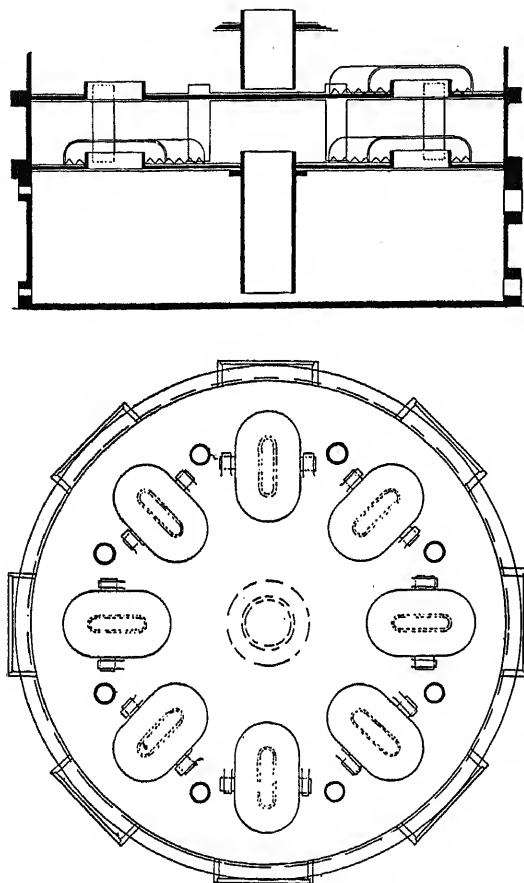


FIG. 64.—ELEMENTS OF GAS LIQUOR STILLS.

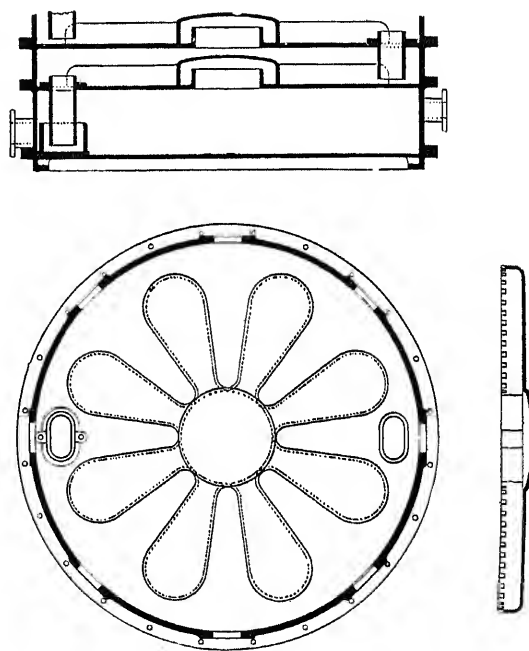


FIG. 65.—ELEMENTS OF GAS LIQUOR STILLS.

the respective bubbles from the several hoods. It will be an advantage if the makers will in future avoid small diameters of the overflow pipes.

Fig. 65 represents the chief constituent parts of a still which was designed nearly forty years ago. While the designer has apparently recognised the importance of maximum agitation of the liquid, it is feared that the area of the serrations, which is largely in excess of the area of the gas outlet, will obviate this desideratum.

Fig. 66 furnishes details of the elements of a square still, certain advantages of which have already been named. Here the importance of vapour velocity appears

to have been recognised, although impact of the bubbles appears impossible. The liquor overflow might with advantage be enlarged.

Fig. 67 represents the details of a still which it is understood is used to a considerable extent in connection with ammoniacal liquor arising from the distillation of shale. The position in which the bubblers lie is varied alternately with each chamber. Thus they cross at right angles. Similarly, the overflows are arranged alternately. This design, it is feared, does not allow of a sufficiently rapid vapour

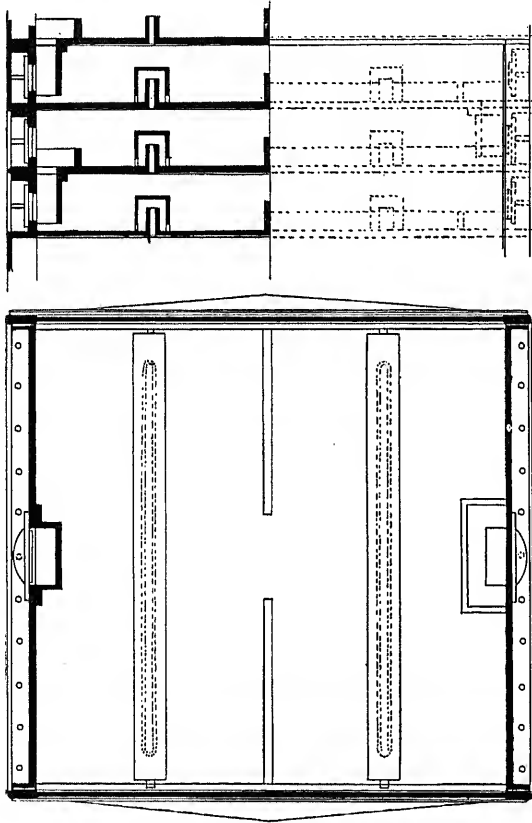


FIG. 66.—ELEMENTS OF GAS LIQUOR STILL.

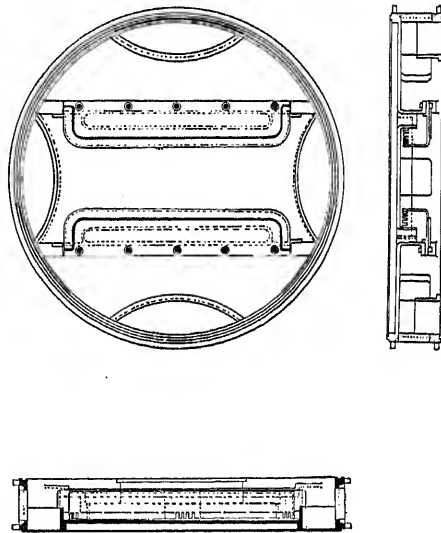


FIG. 67.—ELEMENTS OF GAS LIQUOR STILL.

velocity to ensure satisfactory bubbling, although the arrangement of the hoods and their comparative contiguity will doubtless assist in causing conflict between the respective bubbles.

Fig. 68 is similar in many respects to the elements of one of the other square stills to which reference has already been made.

Fig. 69 represents the sections of a still which has obviously been designed to ensure agitation over a large area, but unfortunately the slow velocity of the gas is not calculated to assist to this end. The manholes appear to afford satisfactory

access to the overflow pipes, but whether the serrations of the hoods can be as easily cleaned, or whether they can be readily withdrawn, is a matter of doubt. If there had been a larger vapour space above the liquid level in the trays, this would have ensured a greater time contact, which obviously is a decided advantage.

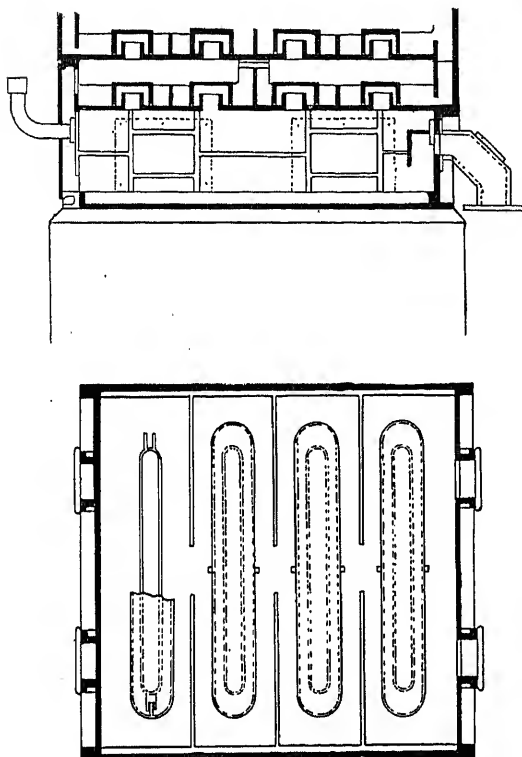


FIG. 68.—ELEMENTS OF GAS LIQUOR STILLS.

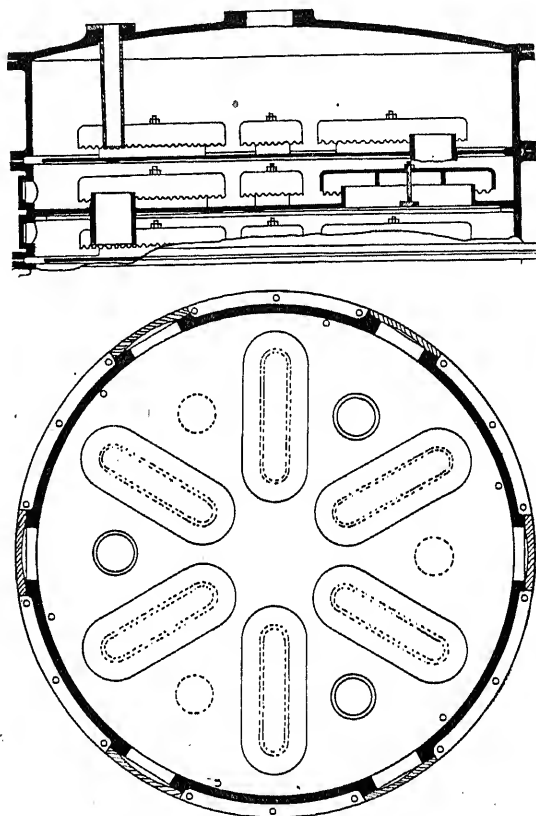


FIG. 69.—ELEMENTS OF GAS LIQUOR STILLS.

RECENT DESIGNS OF STILLS

In a comparatively recent patent Wyld provides for a cascade type of still, the serrated plates (of which fig. 70 shows an isometric projection) are adjustable, and are invariably fixed with the apex of the serrations just out of the liquor. The steam sweeps the surface of the liquor in the troughs and promotes agitation. Deposits are obviated, and fresh surfaces are exposed to the action of the steam-ammonia stream. Irrespective of the foregoing, a portion of the liquor overflowing the weirs is brought across the contiguous trough in the form of a thin spray. An improvement has been introduced as a result of experience of the practical operation of this still. Fig. 71 shows this newer type of still, and fig. 72 is an enlarged view showing the overflow.

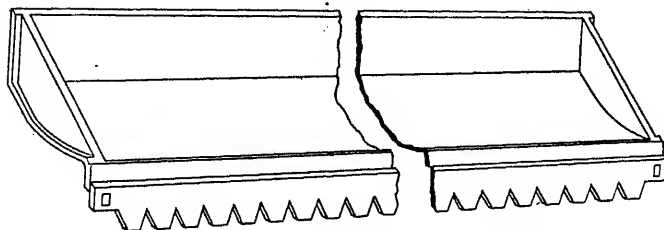


FIG. 70.—SERRATED PLATES FOR CASCADE PLANT.

Messrs. W. C. Holmes, Boocock, and Wyld have taken out a patent, No. 167719, 1921, for a novel type of distilling arrangement. Heated ammoniacal liquor is sprayed in succession into two or more evaporating chambers, the pressure in the first chamber being utilised to effect the spraying in the second. The liquor may be passed through an intermediate chamber (S), in which it is mixed with a reagent to liberate fixed ammonia. The chambers are heated by flue gases circulating in jackets (F). The ammoniacal liquor is preheated by discharge through tubes (K) in the jackets. Connecting tubes serve to pass the gases generated during the heating of the liquor in the tubes (K), to the collecting main (γ), which leads to a saturator for the formation of ammonium sulphate. If pure ammonia is to be separated, the first evaporating chamber is surmounted by a column up which the vapours pass, and down which a portion of the cold ammoniacal liquor flows, and removes ammonia from the vapours. The liquor falls through the perforated top of the steam chamber. The liquor from the base of the chamber is passed through a vessel (S), and then in succession through spraying chambers, from which ammonia gas is withdrawn. Fig. 73 along with the above description will enable one to appreciate the special arrangement in question.

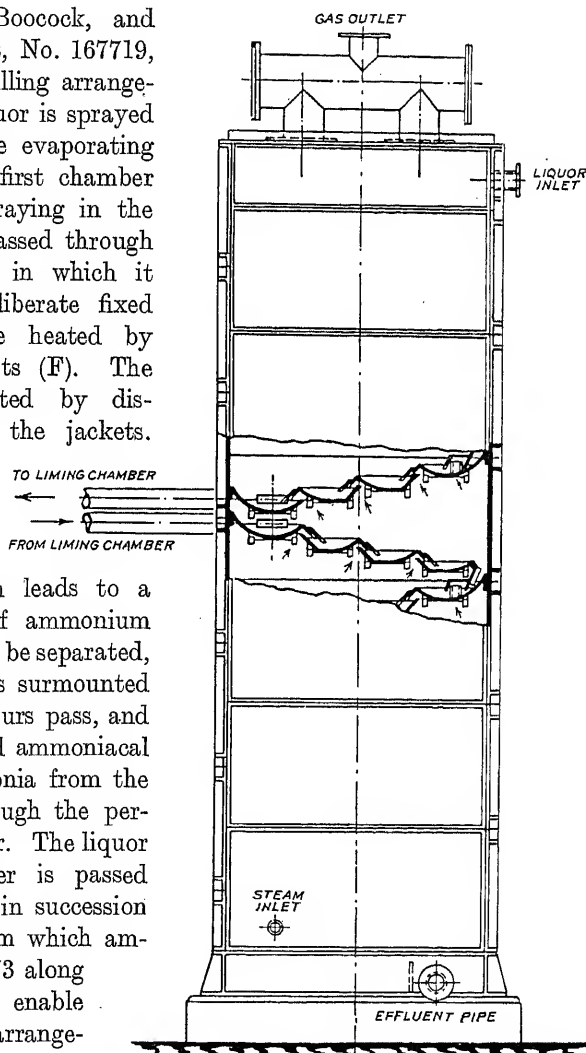


FIG. 71.—WYLD'S CASCADE STILL.

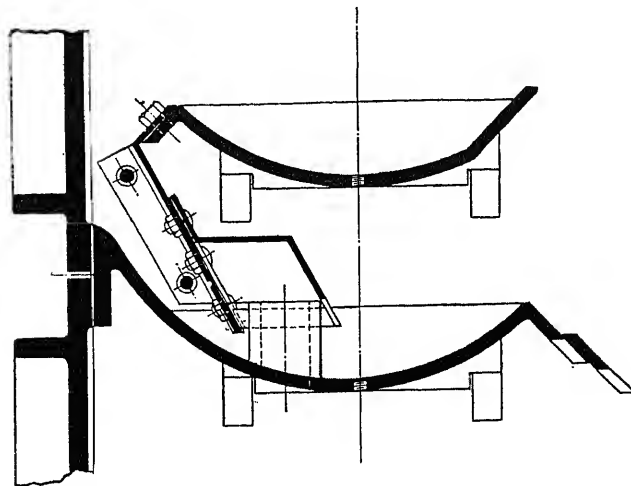


FIG. 72.—ENLARGED VIEW OF WYLD'S CASCADE STILL, SHOWING OVERFLOW.

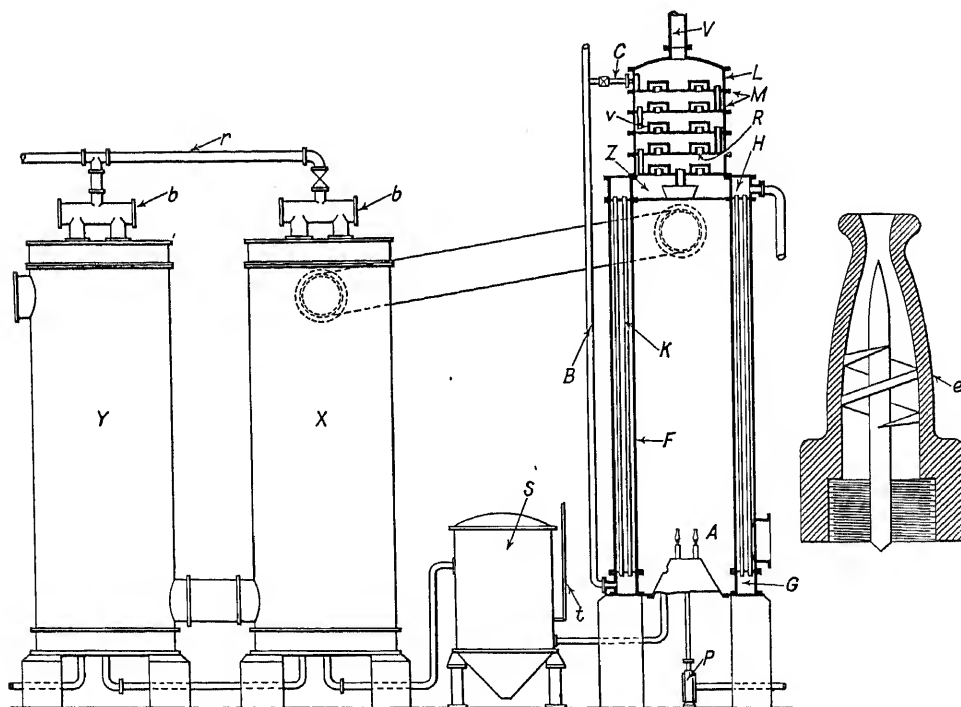


FIG. 73.—JACKETED AMMONIA STILLS, HOLMES, BOOCOCK, AND WYLD TYPE.

Vacuum Distillation

Little has been heard during the last few years concerning the distillation of gas liquor under vacuum. This question, however, was a "live one" more than twenty years ago, when several stills were erected in Scotland by Ernest Scott & Co., Ltd., of London and Glasgow, for the distillation of ammoniacal liquor in the manufacture of sulphate of ammonia under reduced pressure. An interesting paper having reference to this subject was read before the Institution of Gas Engineers on June 11, 1903, by Ballantyne, late engineer and manager of the Hamilton Corporation Gas Department. While the paper, to a large extent, is of a descriptive character, and does not afford much information of a positive technical nature, the view is expressed that the vacuum system of distillation is economical as regards steam consumption, particularly at those works where exhaust steam is available.

It is to be remembered that the utilisation of exhaust steam is not confined to vacuum distillation plants alone. Many gas liquor stills operating under normal pressure conditions are worked to-day with exhaust steam. Obviously, there are advantages by operating under vacuum, inasmuch as the partial pressure of ammonia in the vapour is reduced, and hence equilibrium is more rapidly attained, on account of the ease with which the ammonia is liberated from solution. It will be appreciated that the rate of dissociation of ammonia from its compounds is a function of the difference of the partial pressures of the two systems—liquid and gaseous.

A consideration of the relative vapour pressures of ammonia and water in typical gas liquors at ranges of pressure between (say) 20 in. of mercury absolute and 35 in. absolute leads one to the conclusion that no great saving in the steam consumption is likely to occur, whereas an additional expenditure of steam is required for the operation of the vacuum pump and the pump installed to withdraw the effluent liquor from the still.

Other considerations also operate when working a vacuum still. Some are advantageous, others are the reverse. The volume occupied by steam at (say) 5.7 lb. per sq. in. (the absolute pressure at which the vacuum still at Hamilton is reported to have worked) is 65 cub. ft. per lb., as contrasted with 24.75 cub. ft. per lb. when operating under normal pressure distillation conditions. Thus, under vacuum conditions, the velocity of the steam through the still is appreciably increased, and hence the time contact is reduced correspondingly for a given weight of steam. On the other hand, a greater volume of steam passing over, or bubbling through, the liquor on the several trays is clearly an advantage. Manifestly, the elements constituting the chambers of a still operated under vacuum must have a larger surface area provided for the ascent of the steam and ammonia.

It is believed that one of the difficulties with which Scott & Co., Ltd.—the firm responsible for the introduction of the vacuum ammonia distillation plant—were faced was blockage with lime. Fig. 74 affords details of the Scott's vacuum ammonia distillation plant.

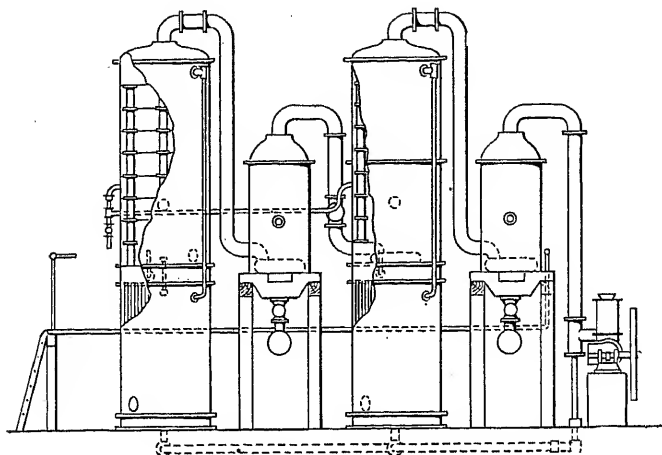


FIG. 74.—SCOTT'S VACUUM STILL.

Methods adopted in the Manufacture of Castings comprising a Complete Ammonia Still, 6 ft. diameter

In view of the large dimensions and cylindrical shape of the main body castings, these are moulded in loam sand. The method pursued is to sweep the outer and inner sections of the mould by means of a spindle carrying a knife-edge board, placed centrally in the bottom ring, on which the mould is built.

The outer and inner parts of the mould, after construction, are thoroughly dried before being finally closed, after which an earthen trough is arranged around the top, into which the metal is poured. This serves simultaneously as a head to give sufficient pressure for the purpose of ensuring a sound casting. Owing to the contraction which takes place immediately the mould is cast (allowance for which is made in its preparation), it is necessary to rip the core from end to end when the iron is sufficiently set, otherwise the strain on castings of this diameter would be such as to cause almost certain fracture. The internal parts of the still, comprising the trays and sundry castings, are moulded in green sand with the use of iron patterns representing the exact shape of the casting required. The usual allowance for contraction is made. Such moulds are only surface dried, and before being closed are blackened in order to prevent the hot metal from burning into the sand. This ensures the castings having a comparatively smooth finish.

Castings of this size, when cold, after being removed from their respective moulds, are put through a patent sand-blast furnace as a means of fettling, the object being to extract the fine particles of sand which would otherwise normally adhere to the surface of the iron. The shells of most large stills are 1 in. thick, and the internal elements vary from $\frac{5}{8}$ in. to $\frac{3}{4}$ in.

CHAPTER VII

THE DESIGN OF DEPHLEGMATORS, COOLERS, AND HEAT INTERCHANGERS

PRIOR to entering into the question of the design of the apparatus constituting the subject of this chapter, it will perhaps be desirable to explain the points at which these various apparatus occur, and subsequently to proceed to a general description, after which the question of design can be considered.

Dephlegmators, which can be regarded as the plant in which an attempt is made to secure condensation at a definite temperature, occur in connection with concentrated ammonia liquor and liquor ammonia plants, and serve the purpose of controlling the concentration of ammonia in the ammonia-steam stream passing to the condensers or absorbers.

In effecting such control they necessarily return water containing ammonia to the still. Such a solution of ammonia is known as the reflux, and obviously the dephlegmator should be so situated in relation to the still that the reflux can gravitate to it through a suitably sealed pipe.

Preheaters or interchange heaters are required at two stages of the sulphate of ammonia process. An interchange of heat should take place between the gas liquor and the waste gases from the saturator, as also between the softened boiler-feed water and the spent liquor. In each case the apparatus must be of a special character, having regard to the different conditions under which they have to operate.

Coolers or condensers are required in connection with concentrated ammonia liquor, liquor ammonia, and sulphate of ammonia plants.

Coolers in connection with the two former processes are required for cooling the steam-ammonia stream to atmospheric temperature, so as to allow of absorption by the water of the ammonia, and so far as the latter is concerned, they are required to cool the waste noxious gases leaving the gas liquor preheaters to atmospheric temperature.

PREHEATERS AND COOLERS

A battery of preheaters and coolers in connection with a unit of sulphate of ammonia plant is shown in fig. 75. The function of the preheater is twofold. As has already been remarked, advantage is taken of the heat of the waste gases and steam leaving the saturator to preheat the ammoniacal liquor prior to entering the still. In this way cooling of the waste gases is partially effected by the gas liquor, the final cooling being completed with water.

With small units of plant it is usual to place the preheater inside the still-house, where it is not subjected to extremes of temperature during operation. With larger units of plant the preheaters are almost invariably placed outside the still-house building. This practice may be regarded as unsound from the point of view of heat conservation. The quantity of heat leaving the saturator, however, is appreciably greater than that required to raise the temperature of the feed liquor to boiling-

point. Indeed, cooling water has to be profusely used to reduce the waste gases to atmospheric temperature.

The thermal aspect of this section of the plant is the subject of further reference (*vide* Chapter IX.), as obviously this is one of the weak points in connection with current practice.

Many forms of preheaters are used throughout the United Kingdom, dependent upon the size of plant for which they are intended, and on the special experience of the maker.

Figs. 76, 77, and 78 show three typical preheaters, each of which possesses distinguishing features.

Heater (fig. 76) consists of a series of double-flanged cast-iron segments, alternate

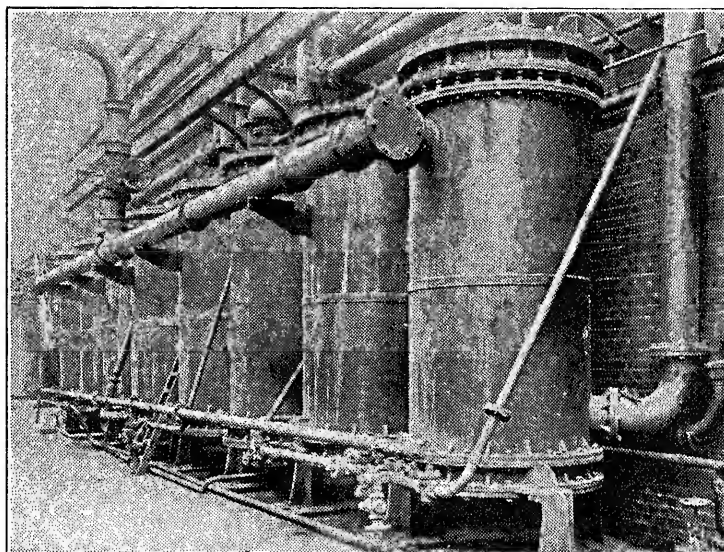


FIG. 75.—PREHEATER AND COOLERS.

ones of which are void, and are provided with flanged inlet and outlet passages, the others being enclosed castings having a series of tubular undulations. The void chambers are coupled together by a series of flanged syphon bends which are placed left-hand and right-hand alternately. The liquor to be preheated enters at *a* and is discharged to the still at *b*. The hot gases from the saturator enter at *c* and leave at *d*. Manholes are provided in connection with each of the enclosed chambers so as to afford access for examination if required. One 4-ft. diameter heater as shown has an effective surface of 634 sq. ft., and is capable of preheating 90 tons of 8-oz. gas liquor per diem. This represents 7.04 sq. ft. per ton of gas liquor per diem.

This type of heater will serve admirably for gas liquor free from tar, but it is suspected that if the gas liquor is of a dirty character the inside of the tubular undulations may become coated with tar, necessitating the dissembling of the sections.

One distinctive feature which this type of heater possesses is the facility and comparative ease with which the segments can be dissembled either for cleaning purposes or for renewal in the event of fractures occurring.

Heater (fig. 77) is of a tubular form, and is arranged to take sixty-one cast-iron

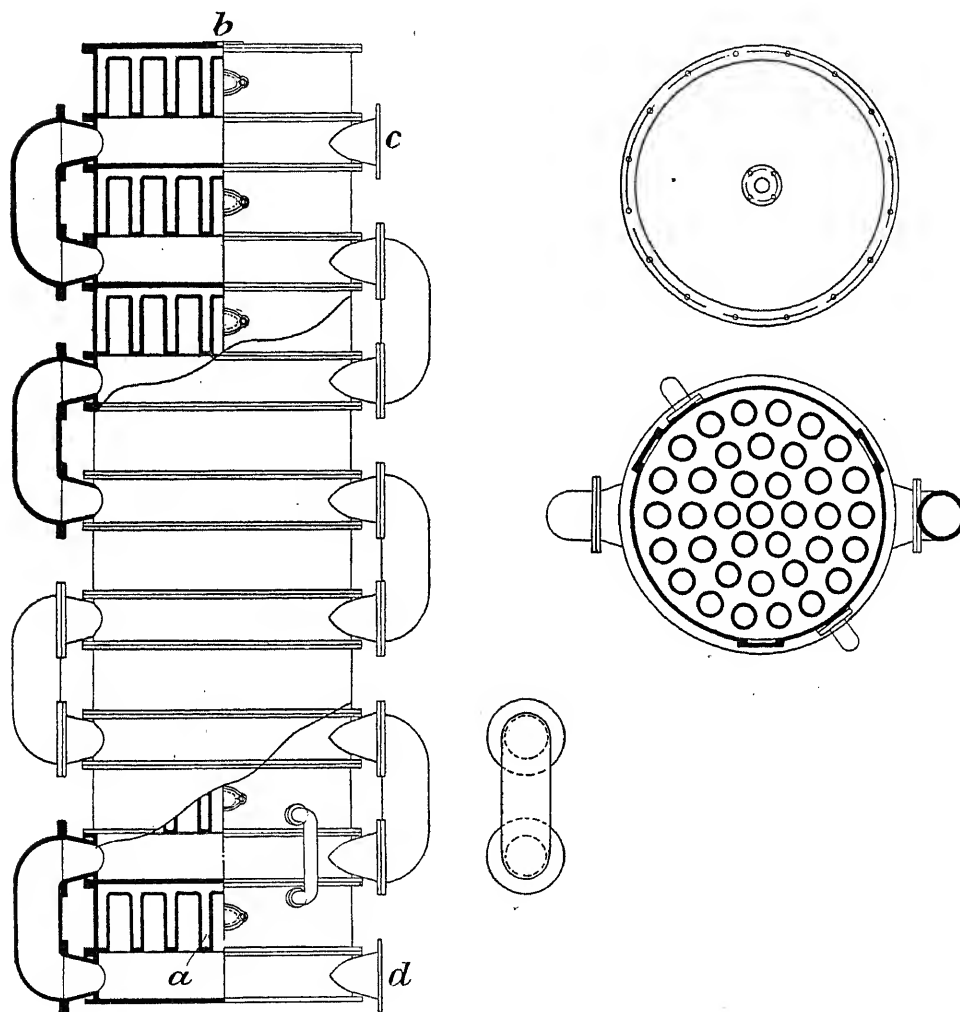
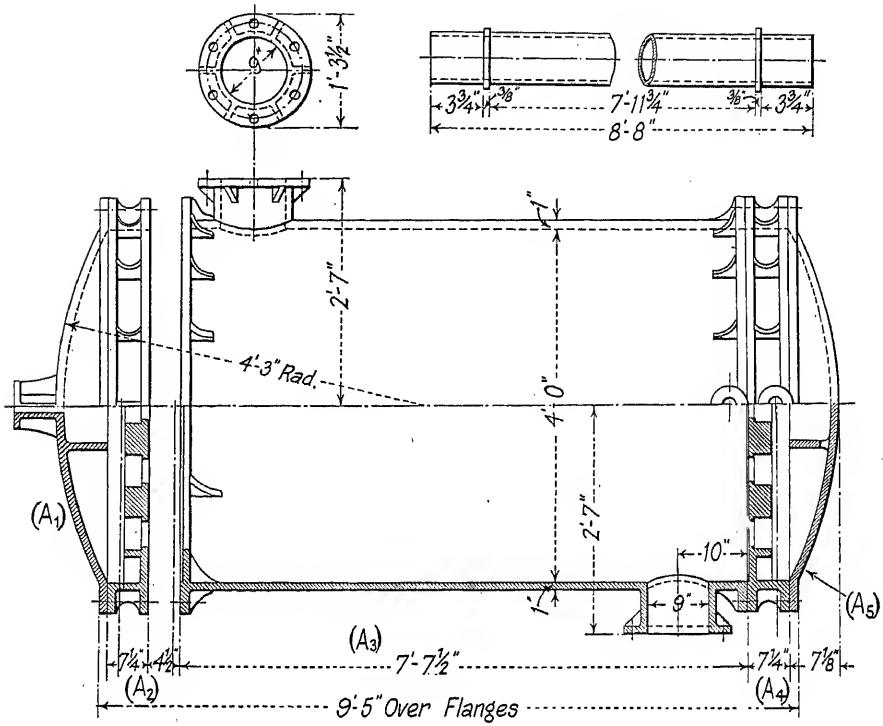


FIG. 76.—HEATERS.

tubes, each 3 in. internal diameter by $3\frac{1}{2}$ in. external diameter. These tubes are provided with shoulders top and bottom, and each fits into suitable recesses in the corresponding tube plates, the annular spaces thus formed being caulked tight with a rust-jointing. To counteract the irregularities of expansion and contraction, as between the shell which externally is exposed to atmospheric influences and the



Half Sectional Elevation

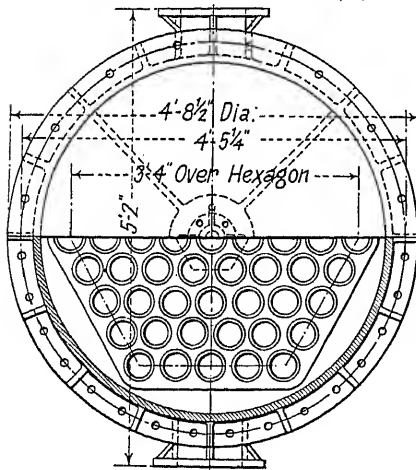
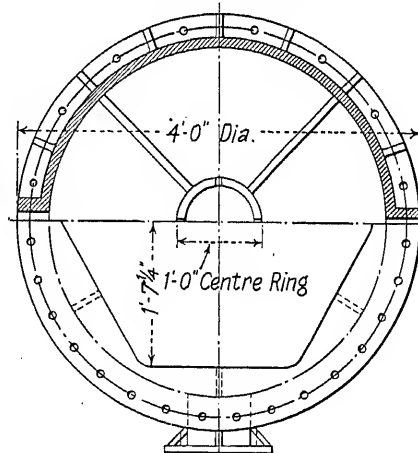
Half Plan of Cover (A₁)Half Plan Segment (A₄)Half Plan of Segment (A₂)Half Plan Segment (A₃)

FIG. 77.—HEATERS.

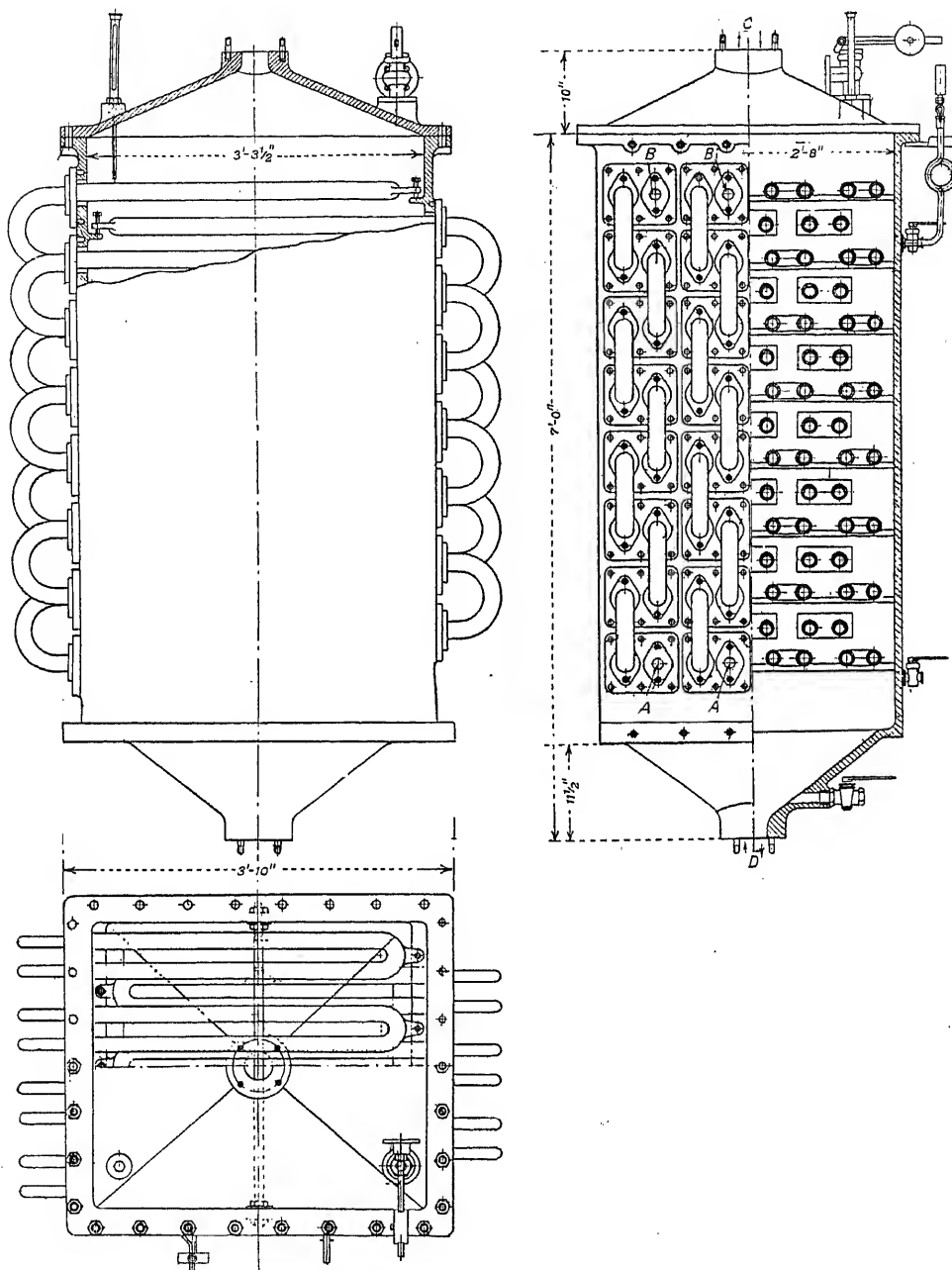


FIG. 78.—HEATERS.

tubes which are in contact with the hot waste gases, a suitable expansion joint D-shaped, made of 32 lb. lead, is provided between the top flange of the heater body

and the tube plate. This arrangement has been found effective in obviating fractures which at one time were experienced with this form of apparatus. The cast-iron tubes constituting the heater must be cast without chaplets, and must be tested to 40 lb. hydraulic pressure.

Fig. 79 shows one of the tubes undergoing the test in question.

It has been found in practice that four vessels of the type represented by fig. 77 are necessary for a still dealing with 30,000 gallons of liquor per day if the gases are to be cooled sufficiently and to be deprived of water vapour to such an extent as to allow of combustion in spent oxide burners. Under such conditions two vessels serve as heaters and two as coolers. The gas liquor is fed to the bottom of the second heater in series and leaves at the top, passing again to the bottom of the first heater

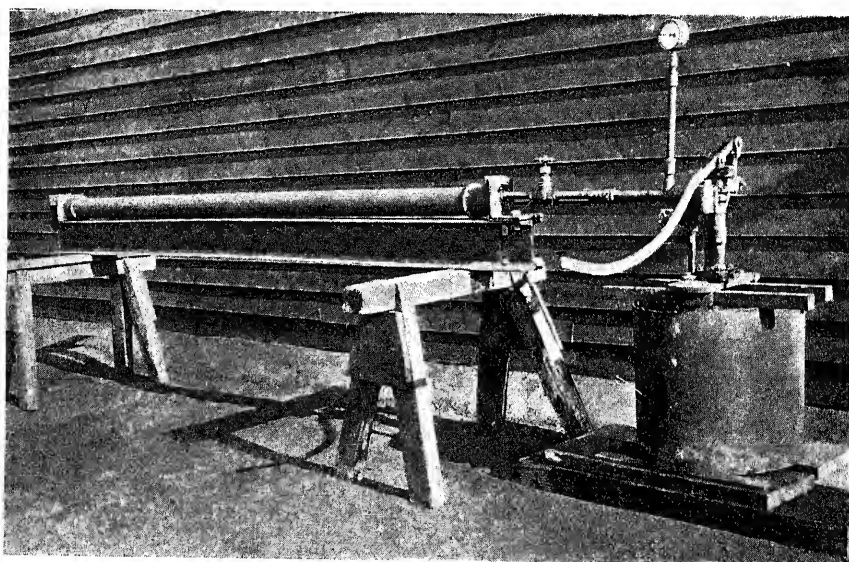


FIG. 79.—TESTING OF C.I. TUBE BY HYDRAULIC PRESSURE.

and leaving at the top to the still. It is important that the liquor should be fed to the bottom in all cases so as to obviate trouble from air locks.

When the vessels serve as coolers it has been found advantageous to insert a circular-drilled cast-iron plate with sixty-one 1-in. to $\frac{1}{4}$ -in. taper holes, so as to ensure uniform distribution of the cooling water. Each heater has an effective surface area of 464 sq. ft. By calculation it will be seen that this represents 6.18 sq. ft. of heating surface per ton of gas liquor per diem.

Heater (fig. 78), which is rectangular in section, has obviously been designed to facilitate replacement of any of the tubes which may fracture or develop leakages without obviating a stoppage of the heater. It consists of seven separate elements, four being placed on one side and three on the other. The waste gases from the saturator enter at C and leave at D. The gas liquor to be preheated enters at seven

different points coinciding with A. This heater affords an effective surface area of 144 sq. ft., and is capable of preheating $14\frac{1}{8}$ tons of 8-oz. liquor, representing 8 sq. ft. per ton of liquor per diem. It is not improbable that this heater is capable of dealing with a larger quantity of liquor than that indicated, as considerable advantage must accrue by reason of the tubes being placed horizontal to the flow of the gases. Such an arrangement admits of a turbulency of motion which is singularly absent in many types of multitubular heaters.

All the preheaters previously described have suitable provision for automatically running the liquid which condenses from the waste gases, and which on account of its disagreeable smell and character has been appositely called "devil liquor." The devil liquor outlets from the preheaters and coolers are suitably sealed to prevent the escape of the waste gases, which consist of hydrocyanic acid, hydrogen sulphide, and carbon dioxide, etc.

APPARATUS FOR SMALL AND MEDIUM-SIZED WORKS

For small and medium-sized works preheaters consisting of two circular double-flanged pieces with a deeper double-flanged piece for the body have been fairly extensively adopted. Fig. 80 shows the type of preheater in question. It is seen that two plates are placed between the top and bottom flanges of the body and the double-flanged pieces. Wrought-iron tubes are used, and are expanded into these tube plates. The author's experience of the use of wrought-iron tubes has not been too satisfactory, owing to corrosion arising at the cooler parts of the tubes, due to the formation of iron sulphide and the influence of the hydrocyanic acid of the waste gases.

Of course it is recognised that a better transmission of heat is possible owing to the thinner walls of the wrought-iron tubes as contrasted with the cast-iron ones, and equally it is appreciated that the greater resiliency and tensile strength of wrought-iron tubes are decided

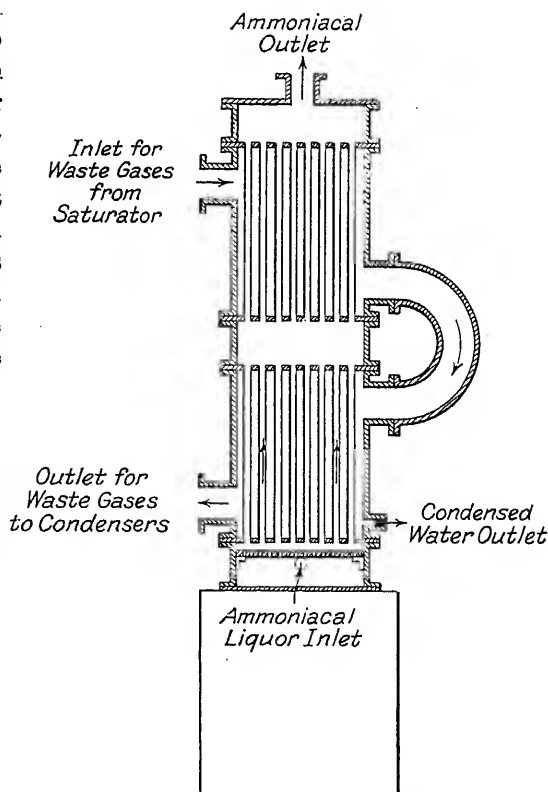


FIG. 80.—PREHEATER FOR SMALL AND MEDIUM-SIZED WORKS.

factors in their favour. Notwithstanding these advantages, the author does not hesitate to recommend cast-iron multitubular preheaters.

The life of any preheater, whether provided with cast-iron or wrought-iron

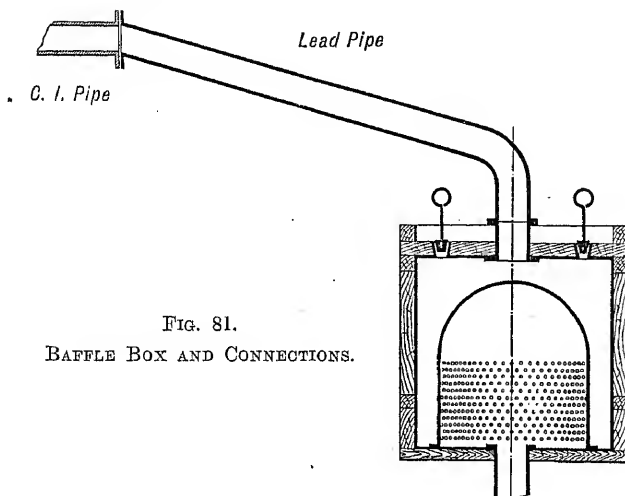


FIG. 81.
BAFFLE BOX AND CONNECTIONS.

pipes, is materially influenced by the freedom with which the waste gases and steam are deprived of vesicular ammonium sulphate solution containing acid, which is mechanically carried forward from the saturator. Cast- or wrought-lead baffle boxes of a suitable form must be provided. Fig. 81 illustrates a common type. It is also an advantage to have the first pipe from the catch boxes in lead, and placed at such an angle that any minute traces of acid mother liquor

arrested can reflux to the baffle box. Such an arrangement is depicted in the above figure.

Another form of cooler which not infrequently forms part of a medium-sized

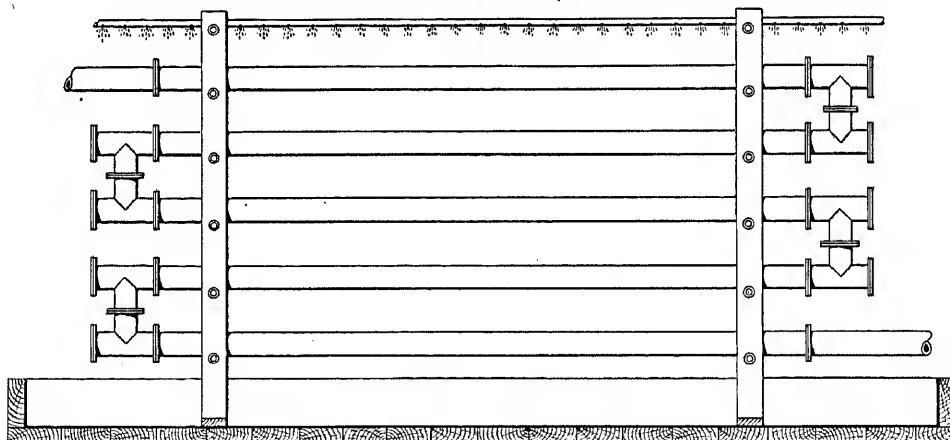


FIG. 82.—COOLER FOR WASTE GASES.

sulphate of ammonia plant is shown in fig. 82. It consists of a series of horizontal pipes connected by means of cast-iron crosses provided with blank plates available for removal in the event of any tarry matter accumulating. The cooling of the serpentine is effected by a spray of cold water, the warm water being collected in a concrete basin and suitably disposed of.

Fig. 83 shows a concentric tube type of cooler which is extremely efficient in operation, affording as it does ideal conditions for rapid heat transfer. It will be appreciated that this design allows of easy cleaning *in situ*.

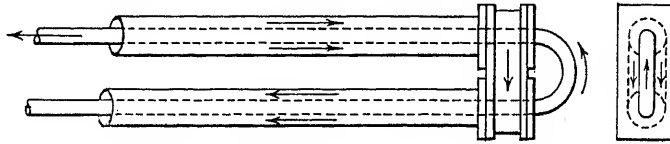


FIG. 83.—SECTION OF COOLER, CONCENTRIC TUBE.

Another extremely well-designed type of cooler which gives a multiple flow for the gas is illustrated in fig. 84. This type of cooler has been found to be highly

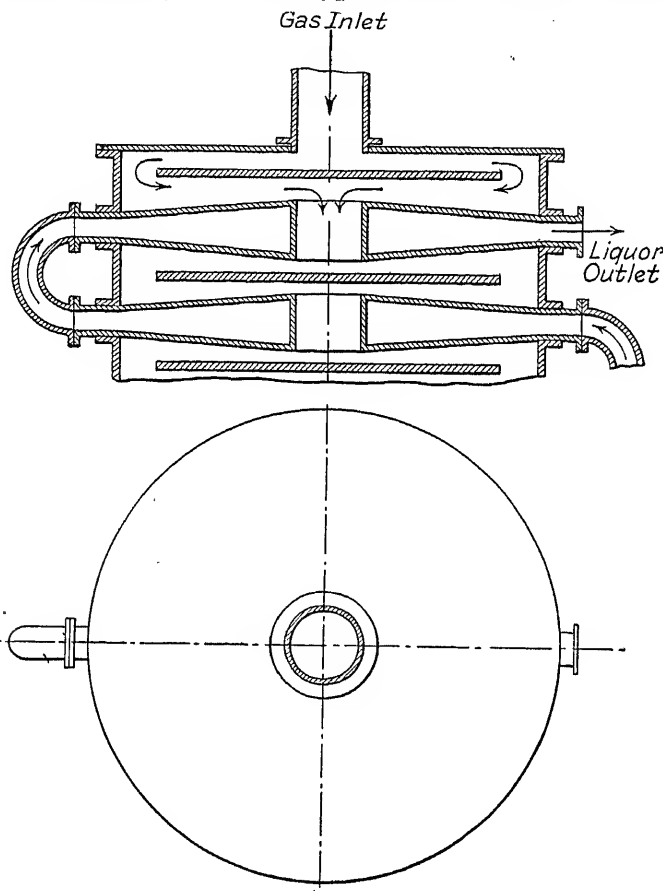


FIG. 84.—SKETCH OF MULTIPLE-FLOW COOLER.

efficient in practice. It possesses the merit of being readily accessible, and it is assembled in such a way as to admit of renewals to any part being carried out readily.

INTERCHANGE HEATER, BOILER FEED WATER

Fig. 85 shows an effective type of heater arrangement for interchange of heat between the boiler feed water and the spent liquor. The chief features which any such interchange heater should embody are these: the spent liquor should pass on the outside of the tubes. These should be arranged as a nest in the respective tube plates

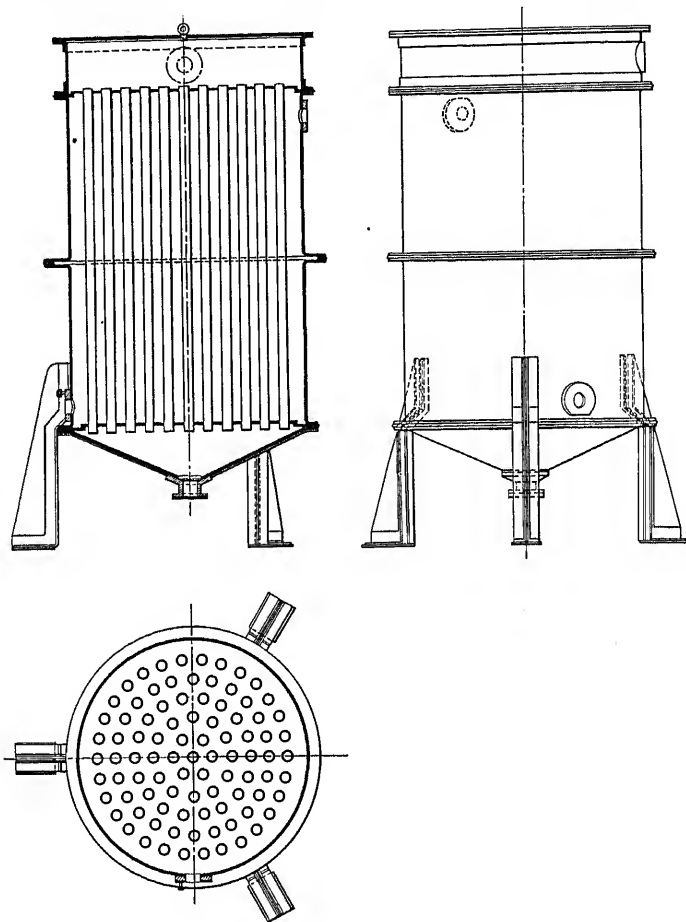


FIG. 85.—BOILER FEED WATER HEATER.

in such a way that they can be lifted out holus-bolus for cleaning. The velocity of the spent liquor should be such as largely to prevent any possibility of the deposition of sludge occurring. In any case, once each week it is preferable to flush the internal parts of the tubes. This work should not occupy more than an hour. Periodically the nest of tubes should be lifted, and any incrustation removed by a special tool. The softened boiler feed water should pass into the heater immediately below the bottom tube plate and should emerge at a point on the body of the heater immediately

above the top tube plate. An expansion joint should be provided, as also a safety valve and vent pipe. The heater should be maintained full of spent liquor by compelling the latter to pass over the "level-maintaining" pipe.

WATER SUPPLY FOR COOLERS AND DEPHLEGMATORS, ETC.

One of the most important considerations on an ammonia distillation works is the provision of an adequate supply of suitable cooling water. At some works sufficient importance is not attached to this question. Town's water is usually too expensive to admit of use, and river water (tidal or otherwise) or canal water has to be resorted to. Unfortunately, this is often pumped through the cooling systems, containing an appreciable amount of solid matter, which invariably, sooner or later, deposits itself at some part or other of the system, occasioning trouble, apart from reducing the efficiency of the coolers. Curiously enough, the mud-freed water is invariably returned to the river or canal.

It is a sound and economic proposition for works to construct a concrete reservoir for the accumulation of such water. The tepid water is pumped through a series of jets so as to form a spray and thus assist efficient air cooling.

In filling such a reservoir and supplying the make-up water, the turbid water should be passed through a pulsometer or other such reverse flow sand filter, such as is illustrated in fig. 86.

A second method of dealing with turbid river water is to pump to a large tank 40 ft. to 50 ft. high, the feed from which is taken off (say) 2 ft. to 2 ft. 6 in. above the bottom of the tank. In this way sedimentation can take place, assuming the area of the tank is sufficiently large, and the supernatant water can be run off to the coolers or other such plant.

Periodically such tank will need to be flushed out to remove the accumulation of mud.

A third and commendable way is the method of the United Water Softeners, Ltd., which virtually combines the two foregoing methods. It consists of sedimentation and subsequent filtration. A series of sedimentation tanks are provided, along with the requisite complement of sand filters of the gravity type.

Milk of lime is used for coagulation purposes, and it is exceedingly important that the lime reagent should be uniformly distributed throughout the water to be treated. The sedimentation tanks should be sufficiently large to afford about three to four hours' time contact for reaction and sedimentation when the plant is operating at its rated capacity. The sedimentation reservoirs should be so designed as to permit of speedy and efficient cleaning.

After the heavier impurities and a sensible portion of the congealed flocculent matter have been precipitated, the water is decanted and suitably fed to the filters.

The filtering medium usually employed is a specially sifted and suitably graded mined quartz sand, supported on a bed of double washed and graded gravel.

As the filter beds become choked they are cleansed with air and wash water. The operation is effected by agitating the filtering medium for about two minutes,

at a pressure of 3 to 5 lb. per square inch. A reverse flow of wash water is then applied to the inside of the filter, and in its passage upwards through the bed the impurities which have been collected on the sand are floated away to suitable collecting troughs.

Operating in this way, each filter is thoroughly cleansed, and can again be put into commission in about twelve minutes.

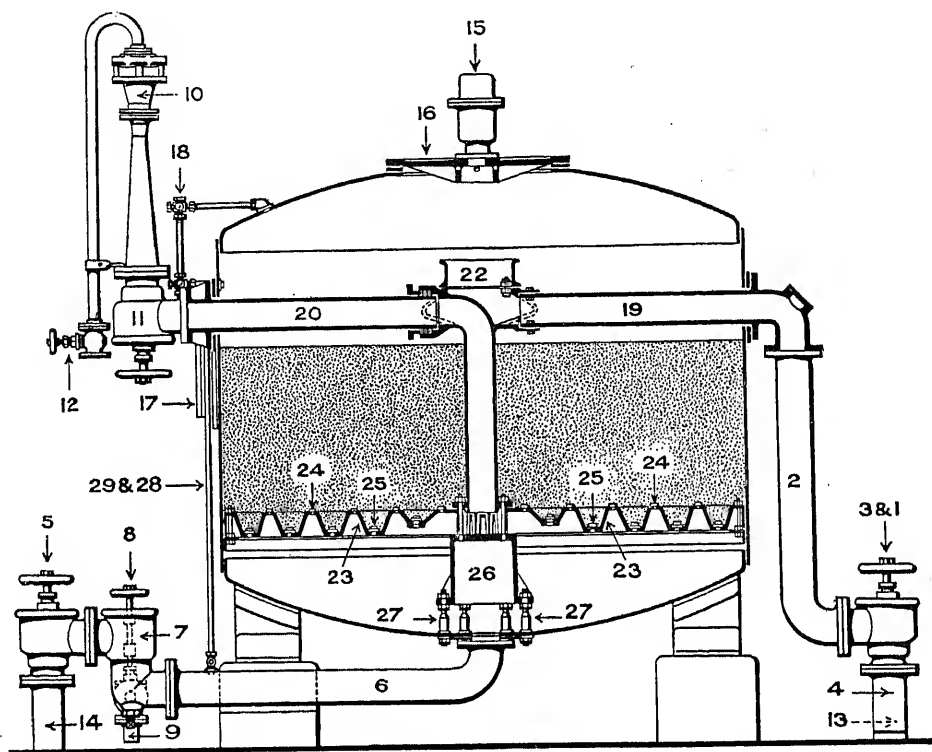


FIG. 86.—PULSOMETER FILTER.

- | | |
|--|--|
| 1. Unfiltered water inlet valve. | 15. Automatic air escape valve. |
| 2. Unfiltered water inlet wash-out bend. | 16. Manhole cover. |
| 3. Wash-out valve. | 17. Differential mercury gauge. |
| 4. Wash-out pipe. | 18. Water gauge glass. |
| 5. Filtered water outlet and wash water inlet valve. | 19. Internal inlet and wash-out pipe. |
| 6. Filtered water and wash water bend. | 20. Internal air pipe to strainer plate. |
| 7. Patent wash water regulating valve. | 22. Internal wash-out and inlet bell mouth. |
| 8. Drain valve. | 23. Strainer plate. |
| 9. Drain pipe. | 24. Air nozzles. |
| 10. Air blower. | 25. Strainers. |
| 11. Blower valve. | 26. Stool for strainer plate. |
| 12. Steam stop valve to air blower. | 27. Studs for strainer plate stool. |
| 13. Unfiltered water pipe. | 28. Pressure pipe from underside of filter medium. |
| 14. Filtered water pipe. | 29. Pressure pipe from above filter medium. |

After the water has been passed through the sedimentation tanks and filters it is collected at a suitable place and pumped through the works to the various points at which filtered water for cooling purposes is required. The cost of such treatment does not exceed 1d. per 1000 gallons, exclusive of pumping costs, and the advantages derived, and the more efficient working secured, justify such a method of treatment. Where 1000 tons of ammoniacal liquor are being distilled per day, it can be counted

that 12,500 to 15,000 gallons of cooling water per hour will be required. If other processes are undertaken at the same works, then even larger quantities will be needed.

SOME CONSIDERATIONS AFFECTING DESIGN

In designing a combined heating-cooling system, such as is necessitated in dealing with the waste gases from a sulphate of ammonia saturator, where part of the heat is used in preheating the gas liquor, the following factors have to be contemplated :

- (1) The amount of heat which must be removed in condensing the waste gases ;
- (2) The amount of heat which must be removed in cooling the condensed steam, etc. ;
- (3) The quantity of vapour entering the cooling system ;
- (4) The amount of inert or other non-condensable gases present in the stream from the saturator ;
- (5) The temperature of the vapour entering the cooler ;
- (6) The temperature of the non-condensable gases leaving the cooling system ;
- (7) The temperature of the condensed vapour leaving the system ;
- (8) The amount of each kind of cooling liquid available, *i.e.* gas liquor and water ;
- (9) The temperature of the cooling liquid available ;
- (10) The temperature to which the gas liquor should be preheated and the temperature at which the cooling liquid should leave the system ;
- (11) The velocity of the gases over the cooling surface ;
- (12) The velocity of the cooling liquid over the cooling surface ;
- (13) The removal of non-condensable gases from the system ;
- (14) The physical characteristics of the condensed vapour (devil liquor) ; and
- (15) The physical characteristics of the cooling liquid.

Perhaps it will be expedient to discuss these various factors and the extent to which the heating-cooling system will be influenced in consequence.

It will be understood that the system must be regarded separately ; firstly, as a heating system, and secondly, as a cooling system. The basis of the design which it is proposed to consider is the treatment of the waste gases from a sulphate of ammonia saturator into which are passed the steam, ammonia, hydrogen sulphide, and carbon dioxide, etc., resulting from the dissociation of 1250 gallons of gas liquor of 1.94 per cent NH_3 per hour, along with the treatment of 294 gallons of devil liquor.

Consideration 1 : The quantity of heat to be removed in condensing the vapour :

$$\begin{aligned} &2940 \text{ lb. steam (Latent heat } 970^1 \text{ B.Th.U. per lb.)} \\ &= 2,850,000 \text{ B.Th.U. per hour.} \end{aligned}$$

Consideration 2 : The quantity of heat to be removed in cooling the devil liquor to the required temperature, (say) 120° F. :

¹ It is known that the latent heat of steam is 971 B.Th.U. per lb. For the purpose of these calculations a round figure of 970 has been taken.

Assuming an initial temperature of 60° F. and a temperature rise of 60° F., then the quantity of water required per hour will be

$$\frac{415850}{60 (\text{temp. diff.}) \times 10 \text{ lb. per gall.}} = 693 \text{ galls.}$$

Considerations 11 and 12 have reference to the velocity of the gases over the cooling surface and the velocity of the cooling liquid over the cooling surface. These aspects will be dealt with later.

Consideration 13 can be ignored in the case under consideration, as the system is under slight pressure (there is a positive flow of gases from the condenser), and hence effective displacement takes place.

Consideration 14: This has reference to the physical characteristics of the devil liquor. Those of importance are: (a) Specific Heat, and (b) Viscosity.

(a) This has been taken into account when dealing with consideration 2.

(b) This may be disregarded, as it may reasonably be assumed that the viscosity is practically identical with that of water.

Consideration 15 affects the design, inasmuch as if river or canal water containing scale-forming impurities is used, then the cooling surface on the water side must be readily available for cleaning.

Regard must now be had to the design of the two systems, and this involves attention to the following points:

TABLE 31

(a) The latent heat of the steam to be condensed is equal to	2,850,000 B.Th.U.
(b) The sensible heat in cooling the devil liquor from this quantity of steam is	278,740 „

hence:

(c) The total heat is	3,128,740 „
---------------------------------	-------------

of which 90.8 per cent is latent and 9.2 per cent is sensible.

(d) The heat required to raise the temperature of the liquor from 60° F. to 191° F. is	2,063,250 „
(e) The heat lost by radiation amounts to	659,000 „

Assuming the radiation loss results in condensation and cooling of the steam (including the H₂S and CO₂), then

3,128,740 - 659,000	= 2,469,740 B.Th.U.
and (f) 2,469,740 - 2,063,250	= 406,490 „

which is the total heat equivalent of the steam leaving the heating system, and represents 12.9 per cent of the total steam, or 404 lb.

Reverting to item (d), and dividing the quantity in the ratio of 90·8 per cent : 9·2 per cent, it is found that 1,873,430 B.Th.U. will be absorbed by the liquor in condensing the steam, and 189,820 B.Th.U. will be absorbed in cooling the condensate to 120° F.

COEFFICIENT OF HEAT TRANSMISSION

The coefficient of heat transmission (K) is constituted of three smaller coefficients, thus :

If k_1 = coefficient of transmission of heat from the vapours to the wall, and k_2 = the coefficient of transmission of heat through the wall, and k_3 = the coefficient of heat transferred from the wall to the cooling medium, then $\frac{1}{K} = \frac{1}{k_1} + \frac{1}{k_2} + \frac{1}{k_3}$.

VALUES OF k_1 , k_2 , AND k_3

(k_1).—For a vapour capable of complete condensation this factor would not be lower than two thousand, while for a non-condensing gas the value would be of the order of ten.

(k_2).—For cast-iron tubes of 0·3 in. thickness the coefficient would be 1600. This is arrived at by dividing the coefficient of heat conductivity of cast-iron (30 B.Th.U. per square foot per degree Fahrenheit temperature difference per hour per foot thickness) by the thickness of the wall (0·025 ft.).

(k_3) is represented by an expression given by C. S. Robinson, which is as follows :

$k_3 = 450 V^{0.75}$, where V is the linear velocity (in feet per second) of the water.

If $V = 0.1$ foot per second, $k_3 = 80$.

If $V = 1$ foot per second, $k_3 = 450$.

If $V = 2$ feet per second, $k_3 = 760$.

If $V = 10$ feet per second, $k_3 = 2500$.

Since the vapours entering the heater contain approximately 80·5 per cent of non-condensable gas, k_1 may be taken as 1000, k_2 has been shown to be 1600 for cast-iron tubes 0·3 in. thick, and k_3 will be 760 if it is assumed for the time being that the water velocity is 2 feet per second.

Therefore

$$\frac{1}{K} = \frac{1}{1000} + \frac{1}{1600} + \frac{1}{760} = 0.00875.$$

$$K = 115.$$

LOGARITHMIC MEAN TEMPERATURE DIFFERENCE

It has been assumed for the purpose of this calculation that the liquor enters at 60° F. and leaves at 190° F., that the vapours enter at 214° F., and that the

devil liquor leaves at 120° F. By taking 9.2 per cent of 130° F. (temp. diff.), it is found that the rise in temperature of the liquor in cooling the condensate is 12° F.; in other words, the liquor will first increase in temperature from 60° to 72° F., and then from 72° to 190° F. during the condensing period.

Thus, basing on the following formula,

$$\ell_m = \frac{\theta a - \theta e}{\log_e \frac{\theta a}{\theta e}},$$

where θa and θe are the temperature differences at each end of the system, one arrives at the logarithmic mean temperature difference during the condensing period as 66.4° F., and during the cooling period as 95° F. The surface required for condensing will therefore be

$$\frac{1,873,430}{115 \times 66.4} = 245 \text{ sq. ft.}$$

Assuming a velocity of 0.1 foot per second for the devil liquor, K is arrived at in the following manner :

$$\frac{1}{K} = \frac{1}{760} + \frac{1}{1600} + \frac{1}{80} = 0.1444.$$

$$K = 70.$$

The surface required for cooling will then be :

$$\frac{189,820}{70 \times 95} = 29 \text{ sq. ft.,}$$

and the total heating surface

$$245 + 29 = 274.$$

The question now arises as to the safety factor which should be adopted in connection with the foregoing computation. It must be borne in mind that the gas liquor to be distilled may vary in strength to the extent of 25 per cent. Hence, a certain flexibility must be afforded. Moreover, it will be appreciated that, due to the presence of tarry matter in the gas liquor, a thin film of pitch will eventually coat the inside of the tubes. Taking these two factors into account, and taking a safety factor of 2, and assuming that 3-in. internal diameter tubes 8 ft. long are to be arranged in two single-flow sections, then it will be seen that 58 such tubes will be required. Hitherto practice has led to the inclusion of 61 tubes per heater. This does not disclose a very wide discrepancy as contrasted with practice, but in connection with the cooling section a marked divergence will be revealed as between the computation and what practice has hitherto dictated.

COOLING SYSTEM

The heat to be dissipated in the cooling section is as follows :

TABLE 32

(1) Condensing water vapour	368,520 B.Th.U.
(2) Cooling the devil liquor	37,970 „
(3) Cooling the gases (to the temperatures already indicated)	9,700 „
Total	<u>416,190 „</u>

The water required for the dissipation of the above quantity of heat is 693 gallons, or 111 cub. ft. The above quantity is based on an inlet temperature of 60° F. and an outlet temperature of 120° F. It appears to have been the practice in the past, in connection with the heating-cooling system, for the coolers to be constructed in the identical manner to the heaters. This observation does not apply to any particular plant. Practically all medium-sized and large installations possess this defect.

Assuming the design of the coolers to be the same as that of the heaters, it is known that there are 61 3-in. internal diameter tubes, representing 3.05 sq. ft. The area of the tube plate minus the total area of the tubes is 8.2 sq. ft. Further, the velocity of the water through the tubes is

$$\frac{111 \text{ cub. ft.}}{3.05 \times 60 \times 60} = 0.0092 \text{ ft. per second.}$$

The next step in the computation is to determine the values for k_1 , k_2 , and k_3 .

(1) *For the condensing period :*

- k_1 : With a mixture of water vapour and uncondensable gases in the proportions indicated the coefficient will not be higher than 100.
- k_2 : If cast-iron pipes with 0.3-in. walls are used, the coefficients will be 1600.
- k_3 : With a water velocity of 0.01 ft. per second, the coefficient has been shown to be 18.

Therefore
$$\frac{1}{K} = \frac{1}{100} + \frac{1}{1600} + \frac{1}{18} = 0.0662.$$

$$K = 15.$$

It will be seen from the above that low low-water velocity affects appreciably the coefficient for the transfer of heat.

(2) *For the cooling period :*

- k_1 : Assuming the velocity is 0.1 ft. per second, this will be 80.
- k_2 will be identical with that for the condensing period.
- k_3 will be much the same as for the condensing period.

Therefore

$$\frac{1}{K} = \frac{1}{80} + \frac{1}{1600} + \frac{1}{18} = .0693.$$

$$K = 14.5.$$

(3) For cooling the uncondensable gases :

k_1 : With a velocity of .01 lb. per second, k_1 will be as low as .5. This figure is arrived at from the following formula due to Weber :

$$k_1 \text{ (in metric units)} = \frac{4.3w^{0.76}C_pS^{0.2}T^{0.5}}{(\text{Mol. wt.})^{0.3}},$$

where w = lb. per sq. ft. sectional area.

C_p = The specific heat at constant pressure.

S = The reciprocal of the hydraulic mean depth in feet (in circular pipes $S = 48/d$, where d is the diameter of pipe in inches).

T = Absolute temperature, ° F.

M = Molecular weight.

Basing on the above formula, the following graph has been prepared, showing the variation of k_1 in B.Th.U. for gas velocities up to 10 lb. per square foot per second. The curves in question are merely comparative, and only apply to the design under consideration.

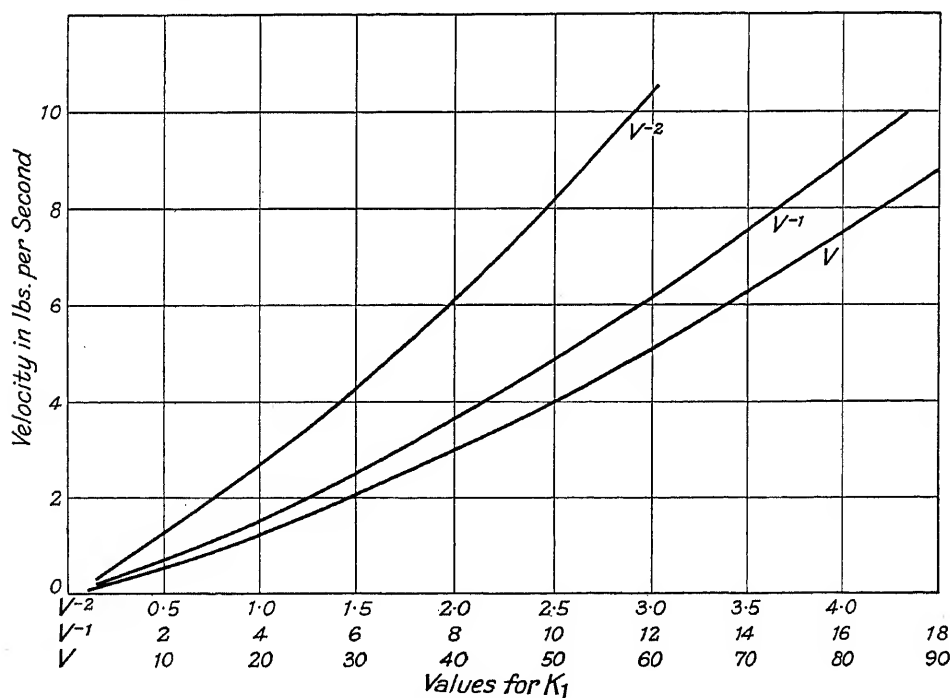


FIG. 87.—GRAPH SHOWING VARIATION OF K_1 WITH VELOCITY.

In this case "gas" implies CO_2 and H_2S in the proportions already indicated under consideration 4, which has reference to the design of heaters. k_2 and k_3 are the same as above.

$$\frac{1}{K} = \frac{1}{0.5} + \frac{1}{1600} + \frac{1}{18} = 2.05618.$$

$$K = .488.$$

Dealing now with the temperature differences in the manner indicated in the calculations for the heater design, the following logarithmic mean temperature differences are arrived at:

TABLE 33

(1) Condensing water vapour	98° F.
(2) Cooling the devil liquor	38° F.
(3) Cooling the gas stream	40° F.

From the foregoing data we can now arrive at the heating surface required.

TABLE 34

(1) Condensing water vapour	$\frac{368,520}{15 \times 98} = 250$ sq. ft.
(2) For cooling devil liquor	$\frac{37,970}{38 \times 14.5} = 69$ "
(3) For cooling the gas stream	$\frac{9,700}{.488 \times 40} = 496$ "
Total	<u><u>815</u></u> "

It is seen that 815 sq. ft. represent the heating surface required in order to perform the work under consideration, but if a safety factor of 2 is contemplated, as is the case with the heaters, then the heating surface to be provided will be 1603 sq. ft. As each of the coolers provides 464 sq. ft., something of the order of four coolers will be required, whereas in actual practice two, with the aid of an atmospheric serpentine cooler, serve the purpose.

The significance of the foregoing calculations is this: they serve to demonstrate that single-flow coolers are by no means satisfactory for functioning under the conditions which have just been considered. Not only is the velocity of the water (if this is to be used economically) through the tubes low, but the velocity of the gases on the outside of the tubes is even worse, from the point of view of heat transfer. Turbulence is out of the question, and the dissipation of the stagnant film on the outside of the tubes is impossible with such a low velocity.

Consideration of this problem reveals the necessity for the cooler system to be arranged on a multiple flow basis if it is to be effective and economical. In point of fact, if one calculates on reasonable velocities for water and reasonable velocities for gas, it will be found that the heating surface afforded by one cooler should be adequate for performing the whole of the work.

It would be outside the scope of this book to give further computations, but, as has been indicated in the introductory chapter, there is an urgent need for the reconsideration of the design of many of the existing types of dephlegmators, coolers, and heat interchange apparatus.

CHAPTER VIII

LIMING, AND THE DESIGN, ETC., OF THE NECESSARY PLANT

As has been explained in Chapter III., ammoniacal liquors contain a certain proportion of their ammonia in the fixed state, thus necessitating the use of an alkali for their decomposition.

Usually caustic lime is converted into a cream or milk by mechanical admixture with water. Occasionally a weak solution of caustic soda, soda ash, or magnesia is employed.

With the latter it is indispensable that it should be recovered, as its cost renders this essential. Numerous endeavours have been made in the direction of the utilisation of magnesia after use in the liberation of the fixed ammonia from ammoniacal liquors, but these do not so much concern the ammonia industry as that of the alkali industry.

PECUNIARY CONSIDERATIONS

As regards the desirability of using caustic soda or soda ash in lieu of lime, the question is essentially a pecuniary one. The present prices (March 1924) for the three materials in question are as follows :

Caustic soda, 76/77 per cent	. . .	£19 7 6	per ton
Soda ash, 58 per cent	. . .	8 12 3	„
Lime, 96 per cent	. . .	1 15 0	„

Approximately 80 parts of caustic soda, 106 parts of soda ash, and 56 parts of lime accomplish an identical amount of work in the liberation of ammonia from fixed ammonium compounds. In the case of caustic soda and soda ash there is no sludge to be dealt with, and, moreover, the cleaning of stills is largely dispensed with. It must, however, be remembered that still cleaning does not cost much more than 2d. per ton of sulphate of ammonia made, and the removal of sludge, even if conveyed to a dump, does not represent more than 2s. 6d. per ton of sulphate of ammonia manufactured.

In the production of one ton of sulphate of ammonia containing 25.5 per cent NH_3 , 0.255 ton of NH_3 is required, or, assuming a 95 per cent working efficiency,

$$\frac{0.255 \times 100}{95} = 0.268 \text{ ton of ammonia.}$$

It will be assumed that 20 per cent of the ammonia of the gas liquor is fixed. Hence, there is 0.0536 ton of fixed ammonia.

$$0.0536 \text{ ton} = 1.072 \text{ cwt.}$$

Knowing this, it is easy to calculate the relative quantities of lime, caustic soda, and soda ash required for the liberation of the fixed ammonia per ton of

sulphate of ammonia manufactured. These respective weights and costs are indicated in the following table :

TABLE 35

Material.	Quantity required per ton of Sulphate of Ammonia of 25·5 per cent NH_3 .	Price per cwt.		Cost per ton of Sulphate of Ammonia of 25·5 per cent NH_3 .		
	Cwt.	s.	d.	£	s.	d.
Lime	1·76	1	9	0	3	1
Caustic soda	2·52	19	4½	2	8	10
Soda ash	3·34	8	7·3	1	8	9

It will be seen that it is very difficult indeed to justify the use of either caustic soda or soda ash in lieu of lime.

“ LIMING ” AND THE LIMING CHAMBER

“ Liming ” has reference to the operation of feeding the cream of lime to the still, and the liming chamber is that portion of the still which is assigned for admixture of the liquor, which has already been denuded of its volatile ammonia, with the cream of lime.

The application of powdered lime by a positive method would undoubtedly prove the most economical, but apparently owing to mechanical considerations this method has not been adopted, except in a few special cases on the Continent.

The advantage of feeding powdered lime to the still resides in the saving of steam and water, the prevention of dilution of the liquor, and the economy of heat due to advantage being taken of the heat of formation of calcium hydroxide. The importance of this aspect is made evident by the data furnished on page 144.

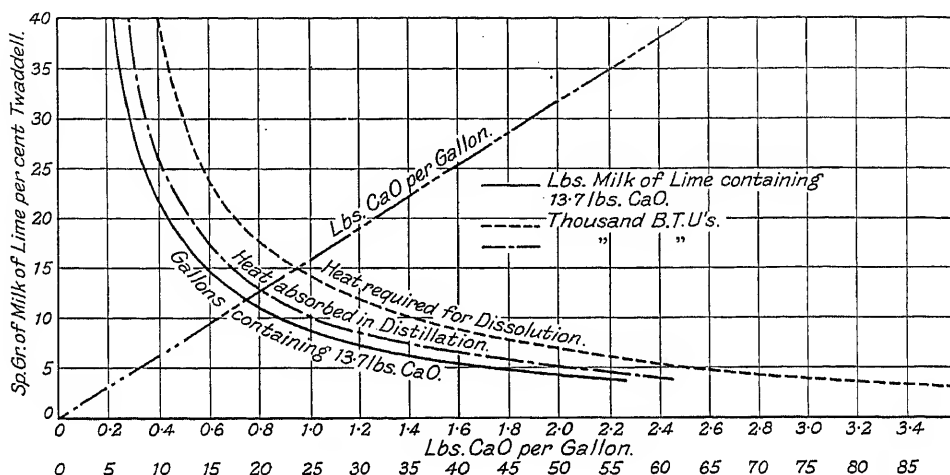
Unfortunately, lime containing 96 per cent CaO and free from ashes is very difficult to obtain, and no doubt this has been one of the deterring factors to the adoption of such a method as the one indicated.

It is found in practice that cream of lime cannot be pumped satisfactorily, and that if pumping is resorted to, milk of lime of about 16° Twaddel at 70°-75° C. has to be used. The lime mixer must obviously be placed in such a position that the cream of lime (say) of 30° Twaddel can be gravitated direct to the liming chamber. When regard is had to the fact that calcium hydroxide is with difficulty soluble in water (only a 0·17 per cent solution at 15° C. being possible), the importance of maintaining the cream of lime in agitation with the liquor need not be unduly emphasised.

The introduction of cream or milk of lime to the still chills the liquor at the foot of the liming chamber momentarily, but sufficient steam, usually about 10 per cent of the total, has to be added to ensure the necessary agitation and to maintain the mixed liquor at boiling temperature.

Coming to the question of the design of a liming chamber, it would appear evident that if agitation is to be efficient there must be a limit to the size of the liming chamber in relation to a given volume of limed liquor treated.

Undoubtedly, one of the faults of design of many liming chambers is that they are too large and allow calcium compounds to settle out instead of being retained



Sp. gr. (° Tw.) . .	4	8	12	16	18	20	22	24	26	28	30	32	34	38	40
Lb. CaO per gall. . .	244	498	752	100	113	126	138	152	164	177	190	203	216	242	255
Galls. required to give 13.7 lb. CaO . .	56.1	27.5	18.2	13.7	12.1	10.9	9.9	9.0	8.4	7.7	7.2	6.7	6.3	5.7	5.4
Lb. required to give 13.7 lb. CaO . .	572	286	193	148	132	120	110	101	95	88	83	78	74	67	65
A. Heat required for dissolution B.Th.U. (1000's) . .	86.4	43.2	29.2	22.4	19.9	18.4	16.6	15.3	14.3	13.3	12.5	11.8	11.2	10.1	9.8
B. Heat absorbed in distillation ditto . .	60.9	30.5	20.5	15.8	14.1	12.8	11.7	10.8	10.1	9.4	8.8	8.3	7.9	7.2	6.9

NOTES.—1. The above figures are calculated for the distillation of 100 galls. of 8-oz. liquor having a fixed ammonia content of 0.346 per cent w/v (20 per cent of the total ammonia).

2. An excess of 20 per cent above theoretical lime quantity is allowed.

3. The milk of lime enters the still at 110° F. and has therefore to be raised through 106.5° to 216.5° (the temperature of distillation).

4. The heat required for the dissolution of the lime is taken as 151 B.Th.U. per lb. of solution.

FIG. 88.—THERMAL ASPECT OF THE APPLICATION OF LIME.

in a state of suspension. This is a matter to which increasing attention could be very profitably devoted.

The question has been asked by managers in charge of small and medium-sized ammonia plants, whether any formulae were known relative to the quantity of milk of lime required per unit volume of gas liquor containing a certain percentage of fixed ammonia in relation to the total ammonia.

Fig. 88 is of interest in this connection. It is clear that a formula of the kind

referred to can be readily calculated from analytical data. One such formula (based on the milk of lime required per hundred gallons of gas liquor) of which the writer has knowledge is expressed thus :

$$V = 65F \div L,$$

where V represents the volume of milk of lime in gallons, F the fixed ammonia content of the gas liquor in oz. strength, and L the strength of lime in degrees Twaddell.

The following calculation will afford an indication as to how the above formula has been arrived at, and incidentally gives an idea of the excess milk of lime solution which it contemplates :

1 oz. sulphuric acid = 0.3469 oz. NH_3 .

1° Tw. milk of lime = 0.845 per cent CaO at 70° C.

Or, in 100 oz. ditto there is 0.845 oz. CaO ;

in 1 gallon ditto there is 1.352 oz. CaO.

$2\text{NH}_3 : \text{CaO}$ as 34 : 56.

1 oz. fixed ammonia requires $\frac{0.3469 \times 56}{34} = 0.5712$ oz. CaO.

The factor per gallon of gas liquor on the above given data is $0.5712 \div 1.352 = 0.42$, or a factor per 100 gallons of 42.

Presumably the formula cited contemplates about a 50 per cent surplus of milk of lime. This is excessive, as extended experience suggests that the whole of the fixed ammonia can be eliminated, given reasonable control, with an excess of milk of lime of 5 per cent of the theoretical.

A useful chart for controlling the consumption of lime in the distillation of gas liquor is shown in Fig. 89. This is reproduced from an article by Harold J. Hailstone, F.C.S., of Rochdale, which appeared in the *Gas Journal*,¹ vol. 161, February 7, 1923. It will be appreciated that it is immaterial whether the ammonium contents are expressed in "percentages" or in "oz. strengths," provided an identical unit is used for both fixed and total ammonia. From the chart in question the correct rate of liquor feed and addition of lime can be readily determined for a given output where liquors of all strengths between 1 oz. and 4 oz. fixed ammonia and 7 oz. to 12 oz. total ammonia are used. Equally, it can be easily adapted for percentage values, and extended to any desired range to suit special local conditions.

To find the amount of liquor and lime required for the production of 1 ton of sulphate from a given liquor it is only necessary to read the total ammonia strength at the side, then trace along to the curve, and from this point read off the gallons on the base line. The lime figure is obtained by tracing the point of total strength from the side until meeting the fixed ammonia line from the base. The point of intersection is read off on the diagonal, and gives the lb. of lime to be used in the distillation of the amount of liquor already found in order to produce one ton of sulphate. For example, for a four-ton per day plant dealing with 10.5 oz. liquor, with 2.8 oz. fixed ammonia, it will be necessary to use 2533 gallons of liquor and

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304 lb. of lime per ton of sulphate produced. This represents 422 gallons of liquor and 50.7 lb. of lime per hour. The chart is based on the amount of lime required to liberate the complete fixed ammonia content from gas liquors of the strengths

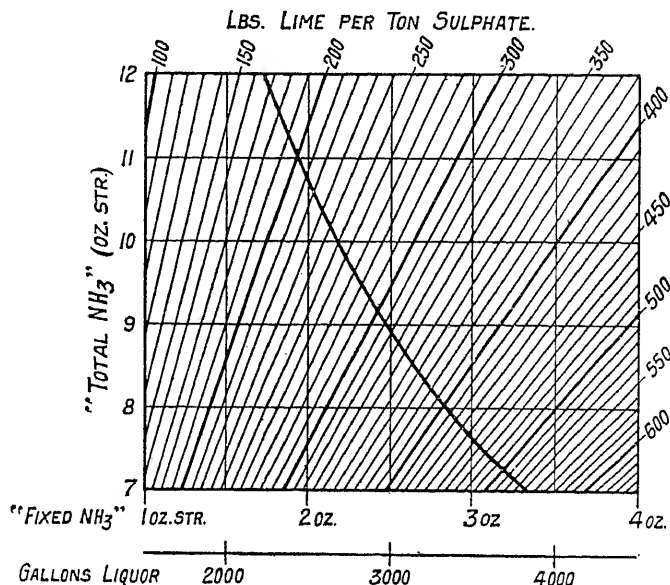


FIG. 89.—HAILSTONE'S CHART FOR CONTROLLING LIME CONSUMPTION.

specified, and provides for a 20 per cent excess to allow for impurities in the lime and for surplus in the distillation.

The lime feed is related strictly to the fixed ammonia content, and, as has been observed, experience suggests that very little excess over the theoretical is required

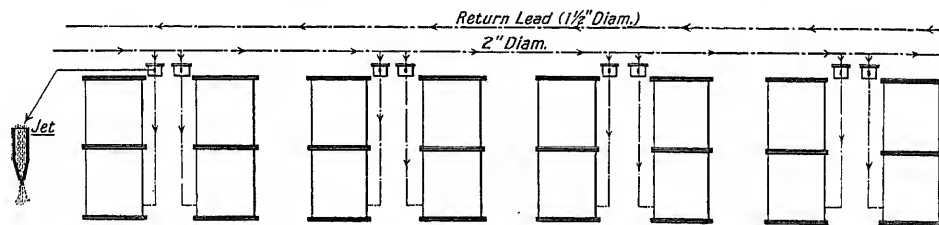


FIG. 90.—SKETCH OF LARGE WORKS LIME FEED.

to liberate the whole of the fixed ammonia prior to discharge of the spent liquor from the still.

An undue excess of lime adversely affects production costs, and leads to increased labour charges in respect of cleaning of the stills and the removal of the sludge from the settling tanks or pits. A deficiency of lime is frequently the cause of a very serious loss of ammonia.

At large works where seven or eight stills have to be fed simultaneously from a

LIMING, AND THE DESIGN, ETC., OF THE NECESSARY PLANT 147

common pipe conveying milk of lime under pressure, it will be found that regulation of the required quantity by a series of cocks is very difficult, and does not allow of a sufficiently sensitive adjustment.

It is preferable to use nozzles with a fine aperture, and to adjust the quantity by increasing or decreasing the pressure in the common feed pipe. In all cases the common feed pipe should be provided with a return having a control cock to the lime tank, to which there must be a continuous discharge. This is illustrated in fig. 90. In this way blockages of the lime service pipes can be obviated.

Deposition of the lime in laminations occurs if the flow of the milk of lime is reduced below a certain velocity.

As illustrating the disparity between practice and theory, the following data, culled from Dr. Bailey's (Chief Alkali Inspector) Annual Report for 1922, are afforded :

TABLE 36
LIME FEED IN RELATION TO FIXED AMMONIA

	Works A.	Works B.	Works C.	Works D.	
	Lime Feed.			Soda Ash Feed.	Equivalent Lime Feed.
Period	6 months	6 months	1 week	2 weeks	
Ammoniacal liquor distilled—volume (galls.)	24,189,000	2,736,000	42,770	29,660	
Fixed ammonia—					
Per cent of total ammonia . . .	23.5	21.0	16.3	23.3	
Total weight (tons) .	441	34	16.2 (cwts.)	23.6 (cwts.)	
Lime as CaO :				As Na ₂ CO ₃ .	As CaO.
Equivalent to fixed NH ₃ (tons) . .	726	56	26.7 (cwts.)	73.5 cwts.	38.8 cwts.
Actually used (tons) .	804	162	28.0 „	93.0 „	49.1 „
Excess used (tons) .	78	106	1.3 (cwts.)	19.5 cwts.	10.3 cwts.
Excess used (per cent)	10.7	190	4.8	26.5	26.5
Used per 1000 galls. of liquor distilled (cwts.) . . .	0.67	1.18	0.65	3.15	1.65
Ammonia in effluent liquor (per cent) .	0.02	0.05	0.02	Average .	0.08
				Maximum .	0.387
				Minimum .	0.014

PLANTS FOR THE PRODUCTION OF MILK OR CREAM OF LIME

Plants for the production of milk or cream of lime are obviously of various sizes according to the capacities of the sulphate of ammonia plants.

Sulphate of ammonia plants range from $\frac{1}{2}$ ton per day to 100 tons per day capacity. With a small plant it is usual to place the liming apparatus adjacent to the still, either in the same building or in a lean-to abutting the still-house. This is necessary, so that the attendant can maintain easy supervision of the whole plant.

With a medium-sized or large works it is desirable, and usually customary, to place the liming apparatus in a separate building adjacent to the lime store.

PLANTS FOR SMALL WORKS

For small works the liming apparatus must be of simple construction, and designed in such a way as to be foolproof.

Consider a works making one ton of sulphate of ammonia per day—and there are many such works throughout the country. It has been shown already that if the fixed ammonia content of the gas liquor is 20 per cent, such a works need not employ appreciably more than 1.67 cwt. of lime per day. Let it be assumed that the milk of lime is to be made of a strength of (say) 12° Tw. This represents approximately 0.752 lb. per gallon.

It will therefore be necessary to prepare for the full twenty-four hours $\frac{196}{0.752}$
 = 261 gallons of milk of lime. This represents approximately 11 gallons per hour, or about one-fifth of a gallon per minute. To feed this quantity regularly throughout the twenty-four hours of the day obviously needs a plant of a foolproof nature.

It is known that in ammonia works of the size under consideration it is the practice to pump a predetermined quantity of milk of lime to the still at certain fixed intervals, (say) each quarter of an hour. The author does not recommend this procedure, as, clearly, there will be a large excess of milk of lime at one time and a deficiency at another. Apart from the foregoing aspect, intermittent feeding of milk of lime chills the contents of the still, increases the flow of liquor through the fixed section, and this means loss of ammonia, as there will be insufficient steam to secure efficient dissociation.

F. Shewring has devoted much attention to the requirements of small works in the matter of lime feed, and has been responsible for evolving one or two special devices for aiding continuous and uniform feed to the still.

The requirements in this connection may be stated as follows :

- (a) A tank provided with a basket, or cage, in which the lime to be treated is placed, and a mixing device by means of which the milk of lime is maintained in a state of constant agitation.
- (b) A small ram pump, or injector, for feeding the milk of lime to a gravity sight feed.
- (c) A special feed device conducting the milk of lime to the pipe leading to the still.

In some cases a tank for slaking the lime is provided, although in the author's

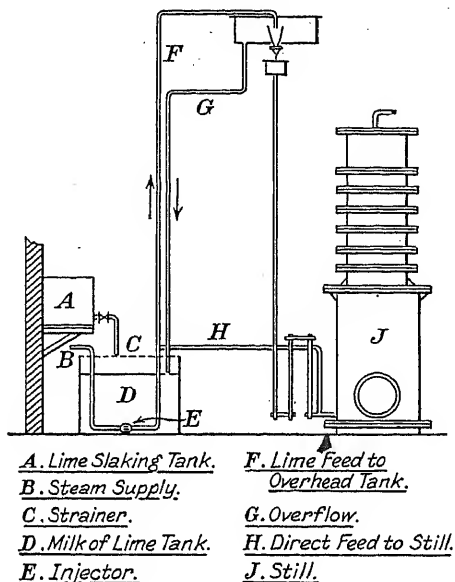


FIG. 91.—SHEWRING LIMING APPARATUS.

view this is not a necessity, as one tank can be made to serve for the slaking of the lime and the maintenance of a suitable suspension as a milk.

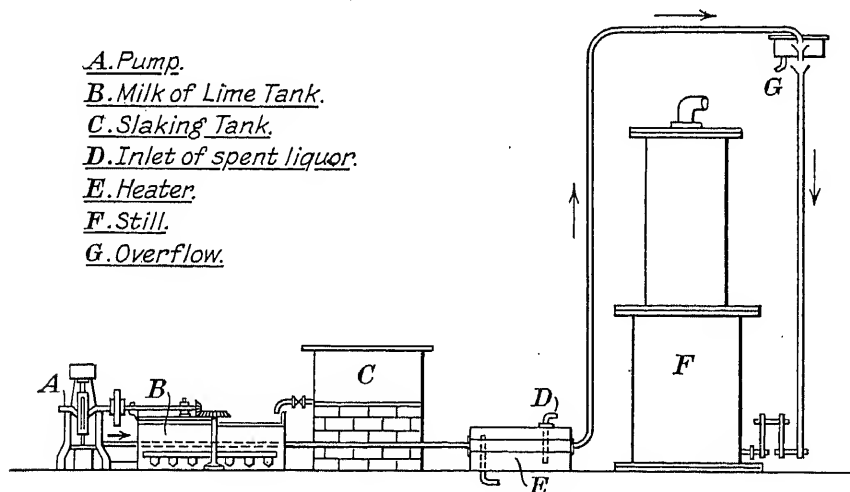


FIG. 92.—SHEWRING LIMING APPARATUS.

Fig. 91 shows a typical arrangement of the Shewring's plant for a small works.

Fig. 92 represents another type of arrangement for the production of milk of lime. Fig. 93 gives details of the Shewring's special lime feed, which aims at avoiding, or reducing the possibility of, blockage of the outlet of the bowl leading to the delivery feed pipe. Vibration is set up in the first case as the milk of lime falls on the light spring. Thus the outlet is continually probed. In the second case the fall of the milk of lime disturbs the equilibrium of the lever arm and automatic probing is secured. The rate of feed for a small plant is so low that it is invariably necessary to arrange for a continuous return from the elevated receptacle to the bottom tank. All the milk of lime fed to the pump should pass through an enclosed strainer having $\frac{3}{8}$ in. diameter perforations, at $\frac{3}{16}$ in. pitch, 18 S.W.G., fixed to the suction pipe of the

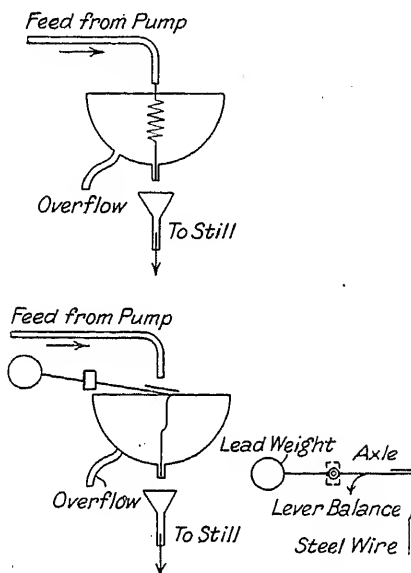


FIG. 93.—SHEWRING LIME FEED.

pump so as to prevent grit, small stones, and ashes reaching the valves of the pump or the nozzle of the injector.

It should be observed that the supply of Shewring's continuous liming device is in the hands of W. C. Holmes & Co., Ltd., of Huddersfield.

MEDIUM-SIZED WORKS

Another arrangement of liming plant suitable for either small or medium-sized works is shown below. This illustration (fig. 94) is largely self-explanatory.

Figs. 95 and 96 represent a complete liming plant as made by Messrs. The Chemical Engineering & Wilton's Patent Furnace Co., Ltd., of London, which has proved efficient in operation. It comprises a lime-slaking tank *a*, with cage *c*, having a partition *b*. Agitation is secured by the operation of a steam injector of

the Korting's or some such type. The steam and induced air are made to pass through suitable perforations in the pipes *d, d*. The grit and ashes remain in the slaking section of the tank and are removed periodically. The milk of lime overflows the

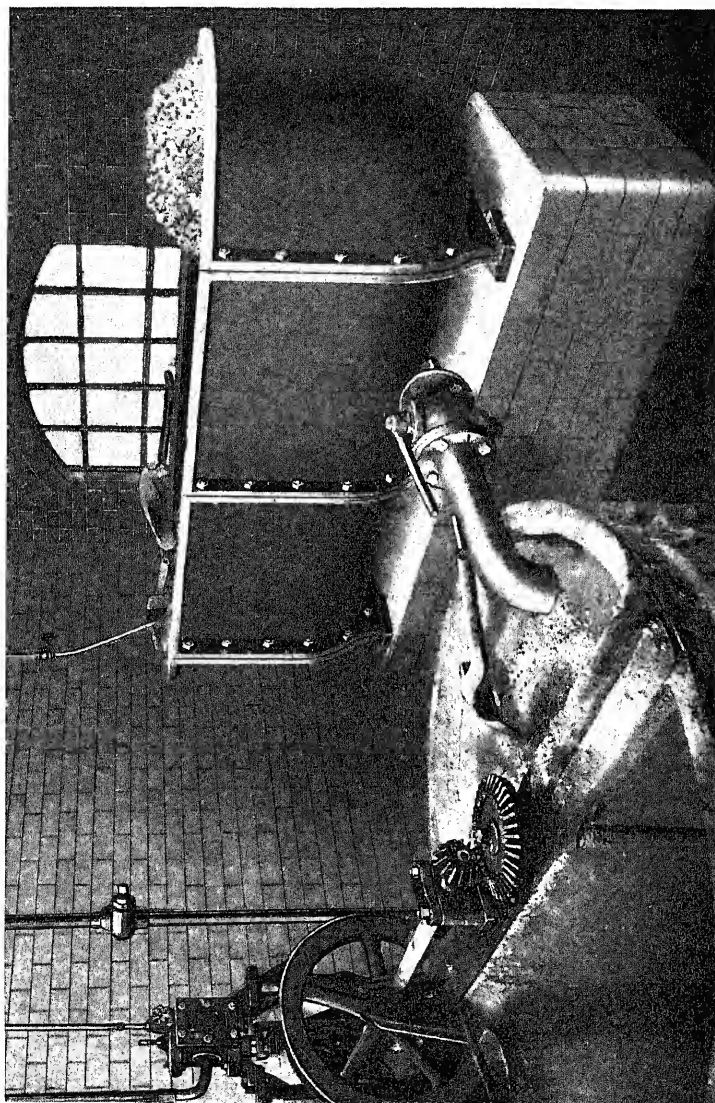


FIG. 94.—LIME APPARATUS.

partition, and is fed to the automatic pumping device. The speed of the lime feed can be regulated by cock *f*, and to obviate any return to the lime tank a check valve *g* is provided.

The automatic pump is provided with a float, which rises as it becomes full.

When full, the rotary valve is thrown over and the admission of steam to the shell

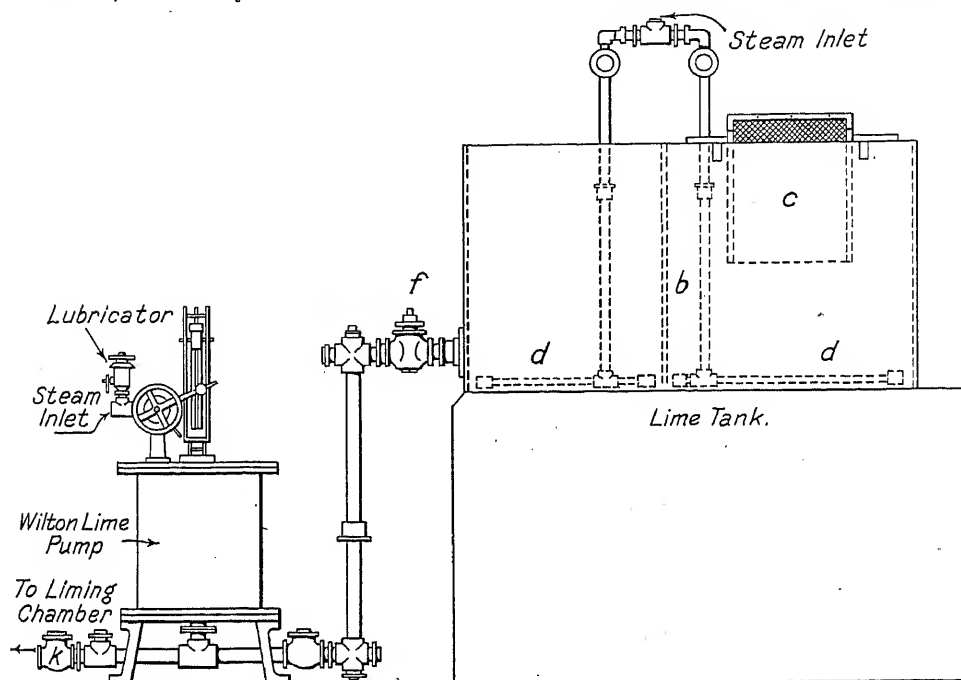


FIG. 95.—WILTON'S LIMING APPARATUS (ELEVATION).

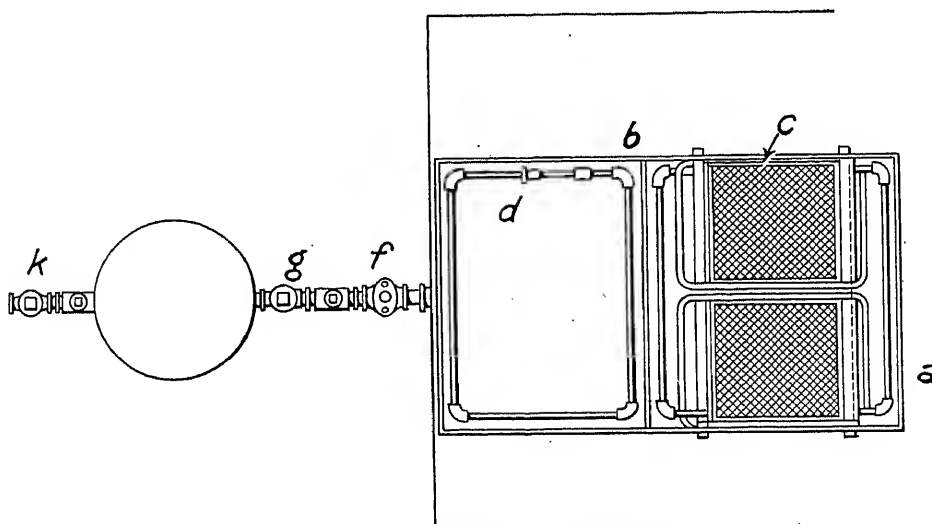


FIG. 96.—WILTON'S LIMING APPARATUS (PLAN).

of the pump is rendered possible. The steam forces the lime from the pump shell

through the check valve *k* to the liming chamber, or to the gravity sight feed. The float gradually descends with the emptying of the body of the pump, and ultimately the rotary valve *i* assumes its original position, when the steam supply is cut out, and the exhaust port is opened, thus admitting of a further cycle of

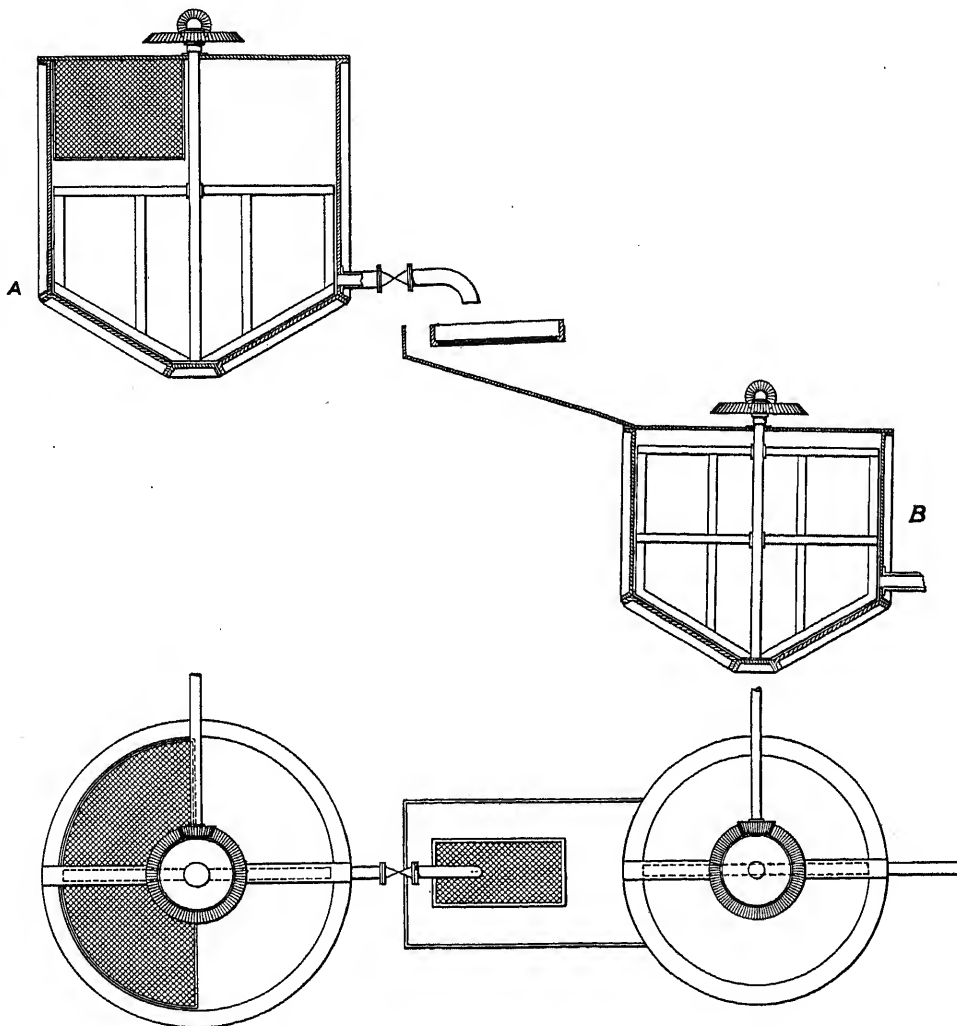


FIG. 97.—ARRANGEMENT FOR PRODUCING CREAM OF LIME 30° TW.

operations. It will be seen that the speed of the pump is not a function of the steam pressure, but rather of the control of cock *f*. A further advantage resides in the fact that no working parts are in contact with the milk of lime, and attrition is largely obviated.

Fig. 97 shows a lime-mixing plant which is specially designed to give a cream of

lime of about 30° Tw. It consists of two conical tanks, each of which is provided with a shaft and agitating arms. The lime is placed in a large basket in vessel A, and this basket is totally immersed in water. Slaking proceeds apace, and the

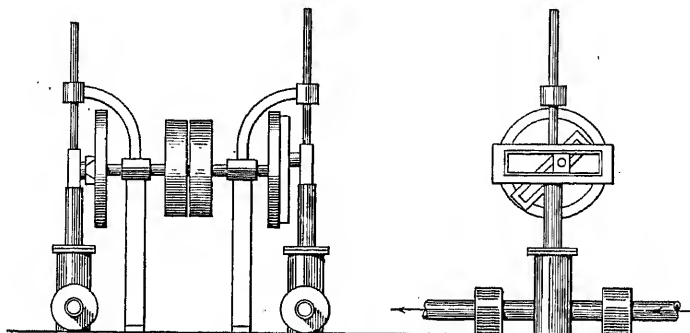


FIG. 98.—LIME PUMP WITH ADJUSTABLE STROKE.

suspension of the lime is aided by efficient stirring. The cream of lime is made in bulk in this vessel, twenty-four hours' supply usually being made at a time. It is then intermittently discharged into a sieve, which allows the man who is replenishing vessel B to push to one side the grit and ashes collected on the sieve, thus admitting of a rapid flow of cream of lime to the second tank.

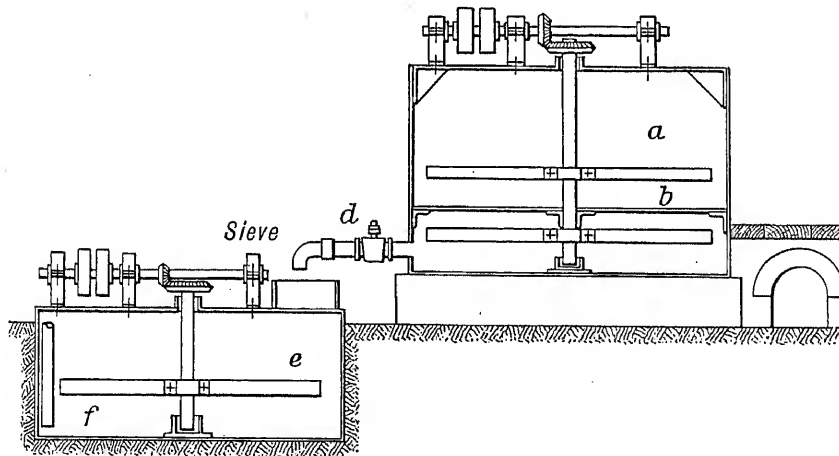


FIG. 99.—DETAILS OF A LIME MIXER.

From the second vessel a special pump is fed with the cream of lime. Details of the pump are given in fig. 98. This pump is a double-acting one, but can be so arranged that one or both sections can be operated. The feature of the pump is that it is driven at a constant speed, which synchronises with the pumping of the gas liquor. Adjustment of the rate of feed of the lime—necessitated by reason of variations in

the percentage of fixed ammonia—is secured by altering the length of stroke of the ram. The crank pin can be fixed at any point on the radius of a special crank disc. It will be seen that the pump is of the plunger type, and is provided with ball valves for the suction and delivery.

LARGE WORKS

A lime-mixing arrangement as shown in fig. 99 is applicable for large works, and will be found to be generally efficient.

Water is run into the circular tank *a*, and the lime disposed on the perforated plates *b*. A hot water or steam supply is necessary to aid the slaking of the lime and facilitate rapid hydration, and the speed of the mechanical stirrer will help the formation of a suspension of lime as a milk or cream. The latter is fed through cock *d*, and *via* the sieve into the tank *e*, from which it is pumped through suction pipe *f*, which should preferably be provided with a closed strainer, to the liming chamber or sight feed. The milk of lime in tank *e* is kept in a state of suspension by a similar agitating device to that provided in tank *a*.

Fig. 100 shows an arrangement of the non-mechanical type for the preparation of large quantities of milk of lime, the agitation of the two tanks being obtained by means of induced air, secured by the operation of Korting's steam agitators. The feature of this arrangement, which is capable of treating 6 tons of lime per twenty-four hours, is the importance which is attached to the removal of grit, etc., from the milk of lime. It will be seen that provision is made for separation of the grit at three distinct points. The conical type of tank, which is provided, also assists the deposition of insoluble matter and aids its removal.

LIMING CHAMBERS

Coming now to the question of typical liming chambers, fig. 101 shows a pipe bringing the gas liquor to be limed to the foot of the still, at which point the milk of lime is added. The steam inlet pipe is provided with four outlets, two of which are placed tangentially, the other two being at right angles to the pipe. The baffle plates admit of considerable travel for ensuring uniformity of admixture.

Fig. 102 represents another form of liming chamber. The liquor to be limed is brought to the foot of the still by four pipes and the milk of lime is admitted at the foot of the still, a suitable agitating steam pipe with radial arms being provided. The operation of the latter is on the principle of the Barker's wheel. The limed liquor ascends the liming chamber and is filtered through a perforated plate prior to being discharged to the fixed still. Whether blockages arise on the perforated plate is not known, but except the agitation is tolerably effective, it is feared that this may prove a possible source of difficulty, particularly so as the perforated plate is not too readily accessible.

Fig. 103 represents another typical liming chamber having many features in common with those already described.

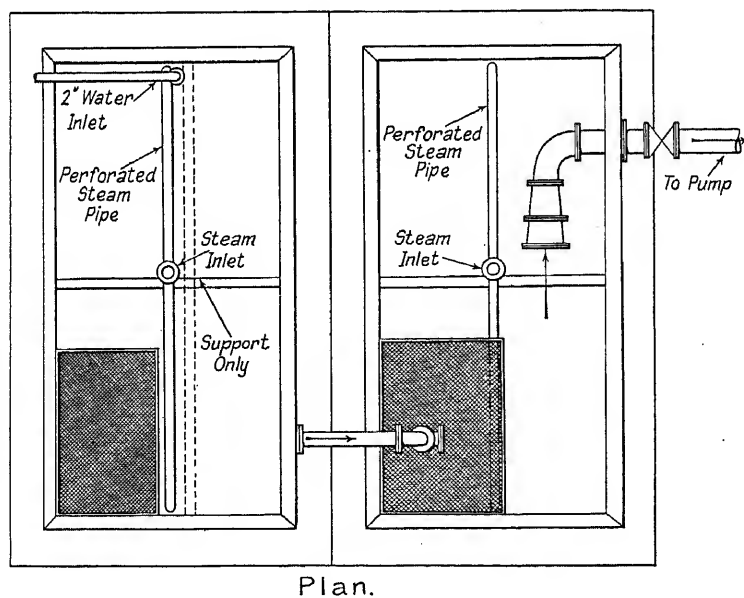
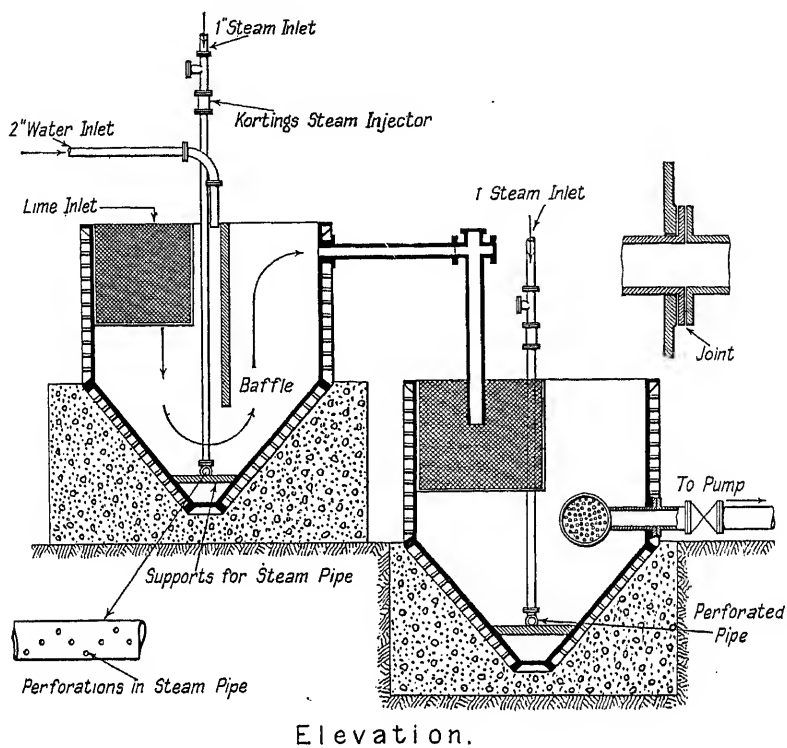


FIG. 100.—LIMING APPARATUS.

LIMING, AND THE DESIGN, ETC., OF THE NECESSARY PLANT 157

It will be seen that this arrangement provides for the limed liquor passing to a chamber at the foot of the column before being led to the fixed still.

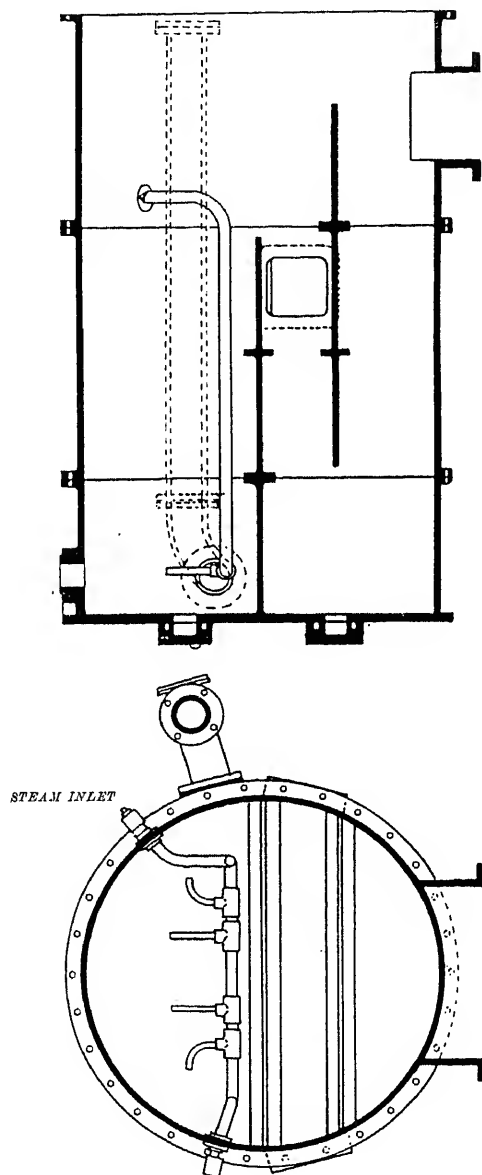


FIG. 101.—LIMING CHAMBERS.

Fig. 104 shows a simple form of liming chamber which intervenes between the

free and fixed stills. The liquor to be limed enters almost at the foot of the chamber, at which point the milk of lime is introduced.

Agitation is created by a circular perforated pipe, the limed liquor leaving by the overflow pipe shown in the sectional elevation.

Fig. 105 shows the liming chamber and stepped fixed still of the Simon's type. This is interesting in many ways.

It is important that the gas liquor should be thoroughly decarbonated before entering the liming chamber, otherwise the ammonium carbonate will react with the

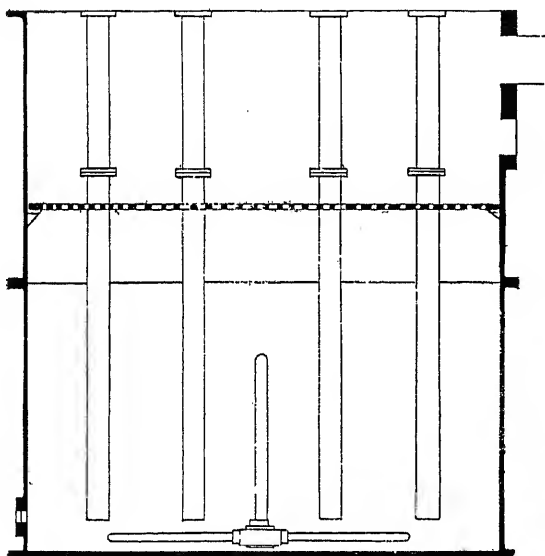


FIG. 102.—LIMING CHAMBERS.

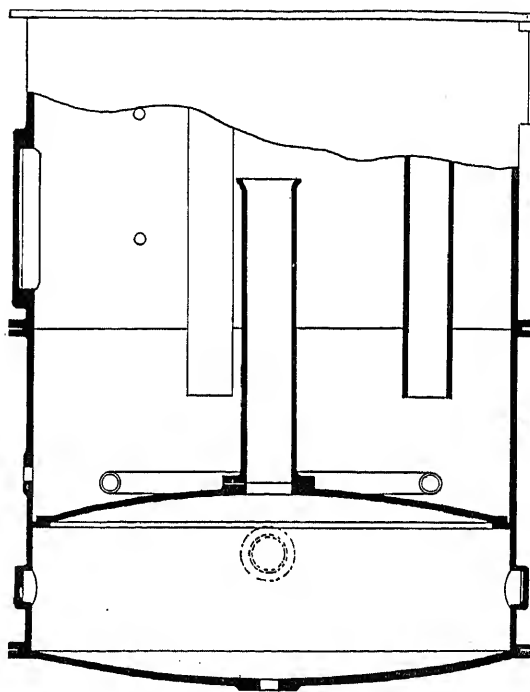


FIG. 103.—LIMING CHAMBERS.

milk of lime, forming calcium carbonate, which becomes hard in the still and renders cleaning much more frequent and difficult.

Further, the amount of lime necessary to effect the removal of the fixed ammonia would of necessity have to be increased, unless an appreciable excess were normally used.

This subject is bound up with the relation of the capacity of the free and fixed sections, which in no known example allows of adjustment of the two sections. Here is a possible weakness which must affect the capacity where the fixed ammonia in the liquor varies. Fortunately, liquor from one source is usually tolerably constant as regards the ratio of free to fixed ammonia : hence this factor would only be of import-

ance in cases where the distiller purchased his liquor from several sources.

Where the liming chambers are of tolerable size, provision should always be made for the periodic emptying of the liming chamber, in order to remove the accumu-

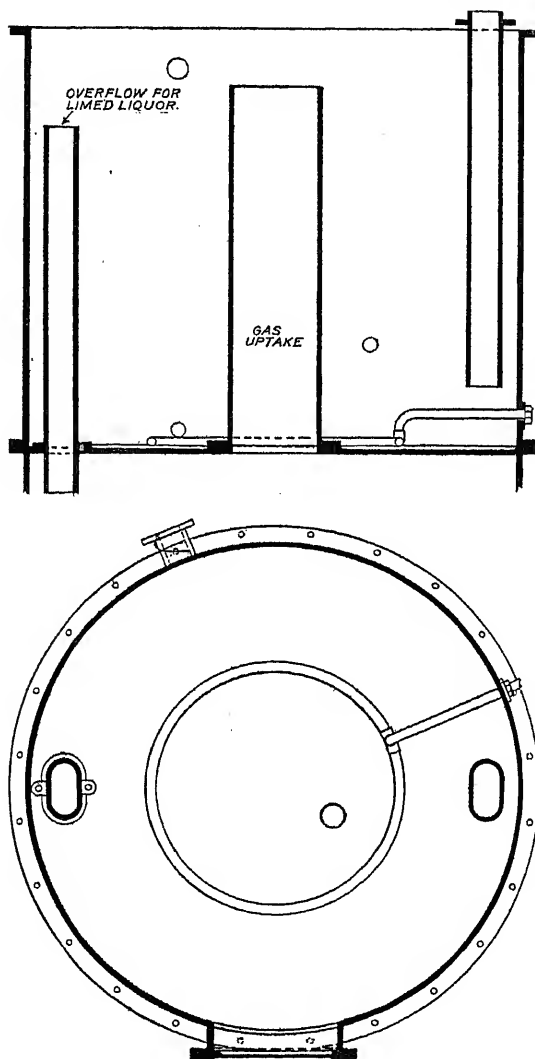


FIG. 104.—LIMING CHAMBERS.

lations of lime sludge which settle out.

Indeed, it has been found advantageous in the operation of many stills to run off part of the limed liquor from the liming chamber direct to the waste liquor main once or twice each day.

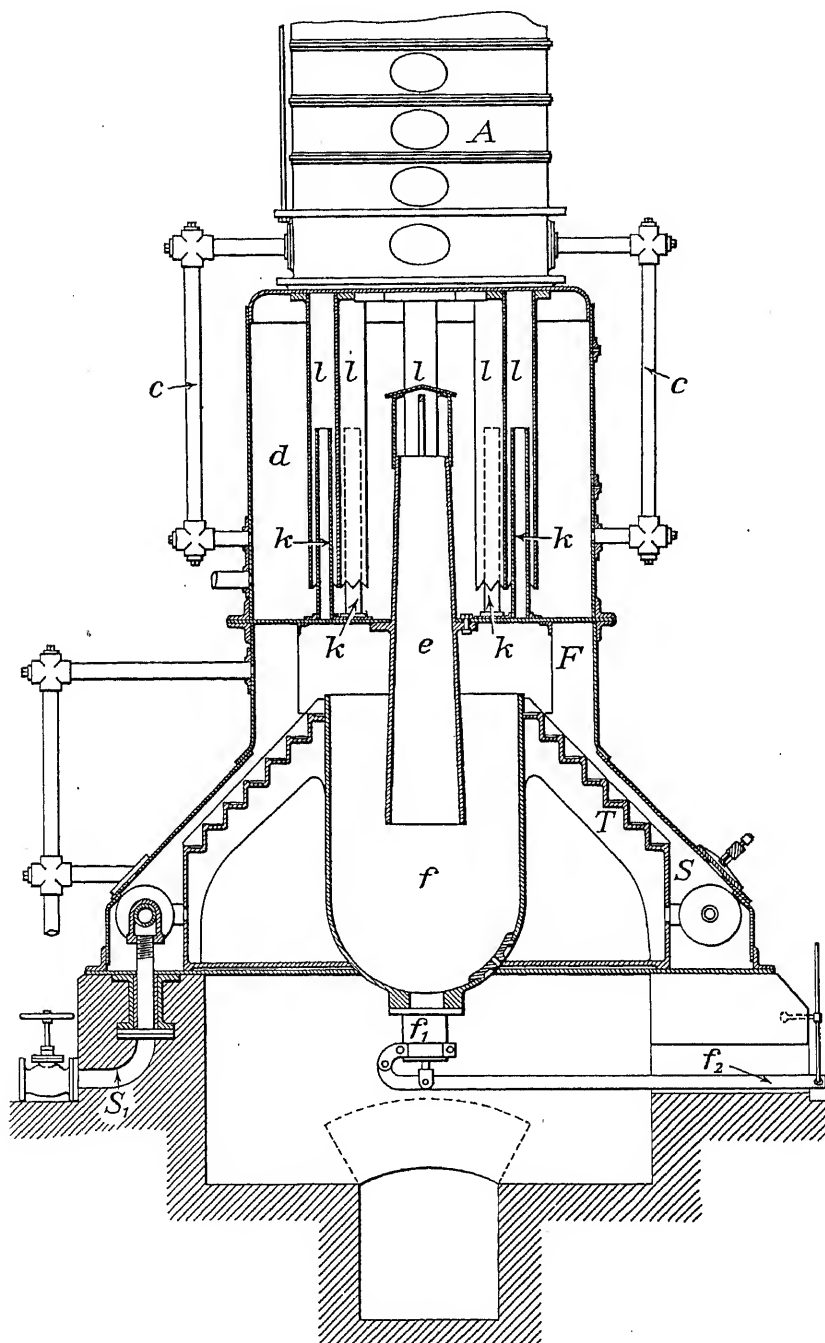


FIG. 105.—SIMON'S STILL. LIMING ARRANGEMENT.

Fig. 106 shows not only the provision for "blowing away" the lime sludge, but the method of operation.

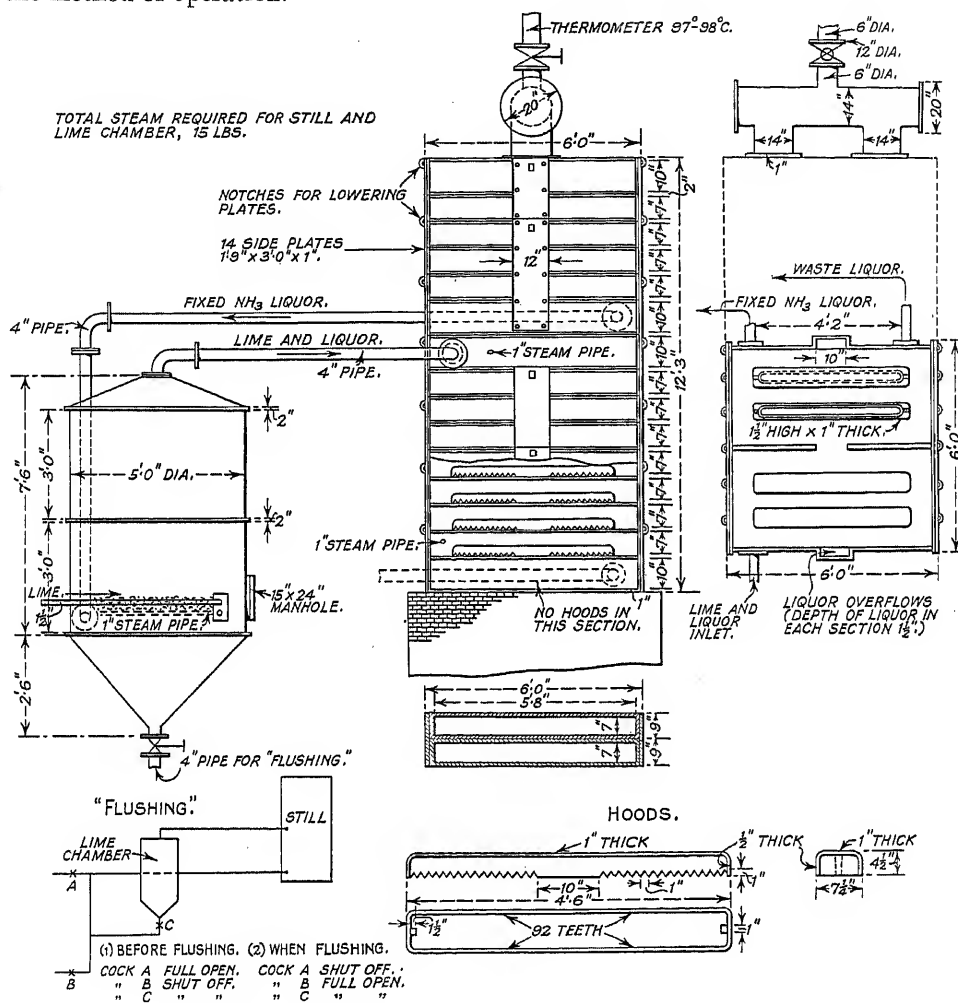


FIG. 106.—SLUDGING, OR "BLOWING DOWN" PROVISION.

Manholes should also be provided in connection with all liming chambers, in order to afford access for cleaning purposes.

As the "blowing down" of ammonia stills results in the loss of ammonia of about 1 to 2 per cent, every step should be taken to reduce the frequency of this operation to a minimum. The greatest importance attaches to the quality and quantity of cream of lime fed to the stills. Obviously, the larger the quantity of grit sent along, the more frequent is the "blowing down." Similarly, the greater the excess volume of cream of lime used, the more often is sludging necessary. At some works the limed liquor which is blown down is collected, and after settlement the clear liquor is pumped to the still and the ammonia recovered.

CHAPTER IX

THE THERMAL ASPECT OF THE SULPHATE OF AMMONIA PROCESS

It has been remarked in the introductory chapter that the greatest economy in the matter of steam, so far as sulphate of ammonia manufacture is concerned, is likely to result from a better utilisation of the available heat of the process.

Hitherto, singularly little information has been published on the subject of the thermal aspect of this manufacture. The author, recognising the importance of this phase, has made many determinations under large-scale working conditions to enable a thermal balance to be drawn up. In this connection the following measurements were made :

- (1) Quantity of liquor distilled.
- (2) Quantity of steam supplied.
- (3) Temperature of steam supplied.
- (4) Pressure of steam supplied.
- (5) Quantity of devil liquor distilled.
- (6) Quantity of cooling water used.
- (7) Temperature of water at the inlet and outlet of the coolers.
- (8) Temperature of the gas liquor at the inlet and outlet of the preheaters.
- (9) Temperature and quantity of cream of lime used.
- (10) Temperature and quantity of effluent liquor discharged from the still.
- (11) Temperature of waste gases leaving the coolers.
- (12) Ammonia, hydrogen sulphide, and carbonic acid contents of the gas liquor distilled.
- (13) Ammonia, hydrogen sulphide, and carbonic acid contents of the gases leaving the still in their passage to the saturator.

The two diagrammatic sketches below, figs. 107 and 108, show clearly the essential apparatus constituting the integral unit, the heat units entering and leaving the various portions of the plant, together with details of the gas liquor being distilled and its content of ammonia. Although the determinations have been expressed on the basis of 100 gallons of liquor (that is, 82 gallons of gas liquor of 1.94 per cent NH_3 content, and 18 gallons of devil liquor, together representing 100 gallons of 1.59 per cent NH_3 content), the actual period during which the measurements were made represented several days.

The various determinations undertaken during the period of the test have been averaged and reduced to a basis of 100 gallons of liquor.

An idea of the capacity of the still and accessory plant on which the measurements were taken can be formed from the rate of feed. This was 100 gallons in four minutes.

The various figures have been summarised, and heat balances are given below. The figures have been calculated from a basis of 15.5°C ., and are expressed respectively in terms of (a) British thermal units, and (b) in pounds of steam from and at 100°C .

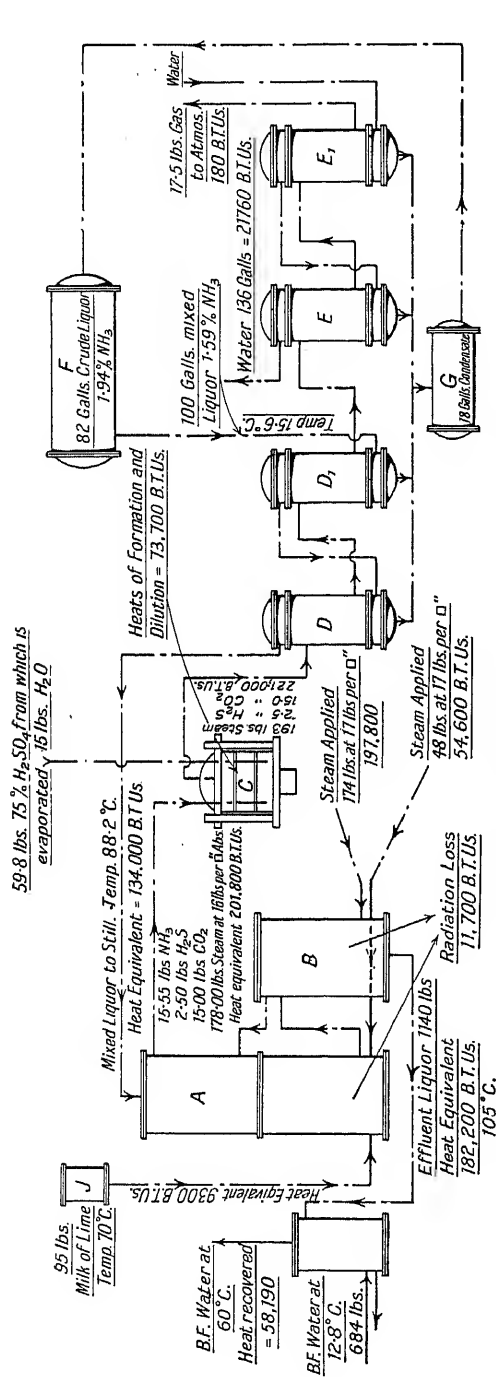


FIG. 107.—THERMAL BALANCE SHEET (1).

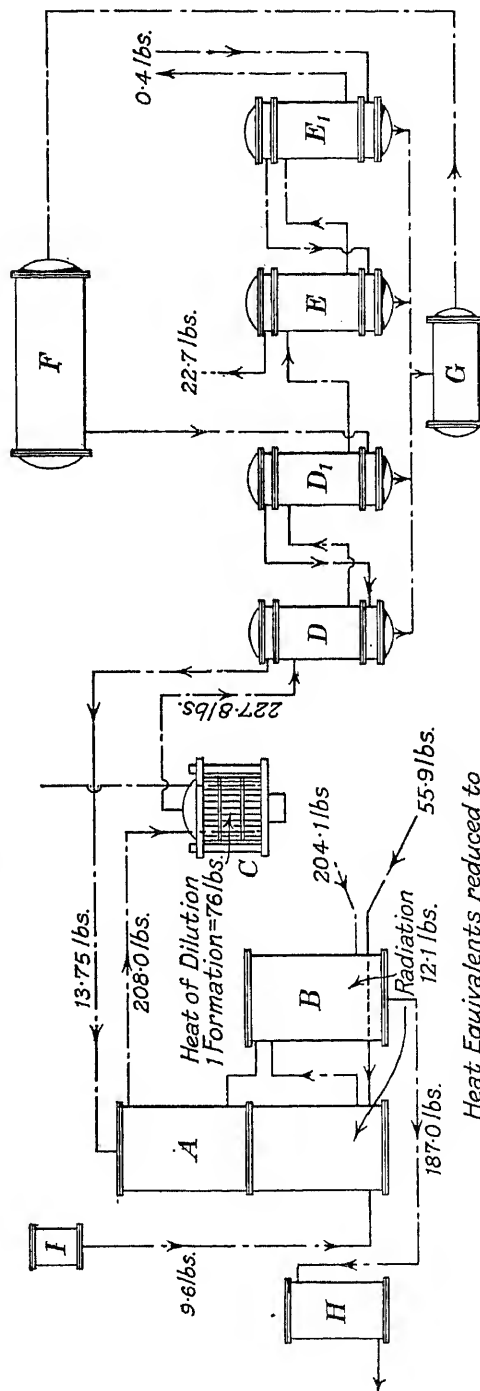


FIG. 108.—THERMAL BALANCE SHEET (2).

- A. Free still.
 B. Fixed still.
 C. Boiler feed water heater.
 D, D₁. Gas liquor preheaters.
 E, E₁. Coolers.
 F. Gas liquor tank.
 G. Devil liquor tank.
 H. Small milk of lime feed tank.
 I. Saturator.

TABLE 37.—SUMMARISED HEAT BALANCE

<i>Dr.</i>		B.Th.U.	<i>Cr.</i>
<i>(a)</i> Heat entering still :			
1. <i>Via</i> steam	Heat leaving still :	B.Th.U.	
2. " liquor	1. <i>Via</i> gas stream	262,400	201,800
3. " lime	2. " effluent *	134,000	182,200
	3. " radiation loss (2.95 per cent)	9,300	11,700
		<u>395,700</u>	<u>395,700</u>
<i>(b)</i> Saturator balance :			
1. Heat entering	Heat entering preheaters	201,800	221,000
2. Heat of dilution and formation	Loss (radiation) during centrifuging, etc. (19.7 per cent)	73,700	54,500
		<u>275,500</u>	<u>275,500</u>
<i>(c)</i> Interchange balance :			
Entering preheaters	Heat recovered <i>via</i> liquor	221,000	134,000
	" lost <i>via</i> water (9.0 per cent)		21,760
	" " gas (0.1 per cent)		180
	Radiation losses (29.0 per cent)		65,060
		<u>221,000</u>	<u>221,000</u>

* Recovery of heat by interchange between effluent liquor and boiler feed water, 58,140.

TABLE 38.—SUMMARISED HEAT BALANCE

<i>Dr.</i>		lb.	<i>Cr.</i>
<i>(a)</i> Heat entering still :			
1. <i>Via</i> steam	Heat leaving still :	lb.	
2. " liquor	1. <i>Via</i> gas stream	208.0	208.0
3. " lime	2. " effluent	187.0	187.0
	3. " radiation loss	12.1	12.1
		<u>407.1</u>	<u>407.1</u>
<i>(b)</i> Saturator balance :			
1. Heat entering	Heat entering preheaters	227.8	227.8
2. Heat of dilution and formation	Loss "radiation" <i>via</i> liquor during centrifuging, etc.	56.2	56.2
		<u>284.0</u>	<u>284.0</u>
<i>(c)</i> Interchange balance :			
Entering preheaters	Heat recovered <i>via</i> liquor	137.5	137.5
	" lost <i>via</i> water	22.7	22.7
	" " gas	0.4	0.4
	Radiation losses	67.2	67.2
		<u>227.8</u>	<u>227.8</u>

THERMAL ASPECT OF THE SULPHATE OF AMMONIA PROCESS 165

Consideration of the thermal balances reveals that the distillation of gas liquor is self-supporting, assuming all available heat is completely utilised. The figures show that in the distillation of the 100 gallons in question the heat entering the still is as follows :

TABLE 39

	lb.
(1) <i>Via</i> steam supplied	260.0
(2) <i>Via</i> preheated gas liquor	137.5
(3) <i>Via</i> cream of lime	9.6
	<u>407.1</u>

The heat available for utilisation is as follows :

TABLE 40

	lb.
(1) <i>Via</i> gas stream from still	208.0
(2) <i>Via</i> effluent liquor	187.0
(3) Heat arising from saturator :	
Heat of dilution and heat of formation	76.0
	<u>471.0</u>

The ratio of steam applied to liquor distilled is 26.0 : 100, or, expressed in another way, 16.35 lb. of steam are required per lb. of ammonia dissociated, from gas liquor of 1.59 per cent NH_3 .

Incidentally, the figures show, assuming no attempt is made to utilise the heat available other than that due to preheating the gas liquor, and ignoring for the time being the work effected by the steam-ammonia vapours leaving the still in evaporating 15 lb. of water, that the efficiency of the process, from the point of view of heat conservation, is 29.2 per cent.

It is unnecessary here to refer to the thermal efficiency of the still *per se*. This aspect has been dealt with fully in Chapter VI.

It is, however, opportune and desirable at this juncture to inquire how best advantage can be taken of the heat available in the sulphate of ammonia process. Although this book is intended to deal essentially with the design and working of ammonia stills, it will be appreciated that one cannot divorce the still from the other portions of the plant to which it is attached when considering heat utilisation.

If, therefore, one is to indicate possible directions for improvement, the plant must be regarded as a whole, and not as a part.

An examination of the thermal balance will reveal that the major portion of the heat is contained in the waste gases leaving the saturator. It is true that part of this heat is utilised in preheating the gas liquor prior to its entering the still. The extent of this utilisation is 60 per cent. The other 40 per cent is dissipated, because it is necessary to use water to cool the gases leaving the gas liquor preheaters to such a temperature as will admit of these being sent, in the case of the plant under

consideration, to the spent oxide burners for combustion to sulphur dioxide. If the gases were not cooled to atmospheric temperature, water vapour would be carried forward to the spent oxide burners, and difficulty would arise in the combustion of the gases, apart from the seal pots of the waste gas main constantly overflowing and causing a nuisance.

It will be seen that the disability with the existing arrangement of sulphate of ammonia plant as invariably erected in this country lies in the fact that there is a larger quantity of heat leaving the saturator than can be adequately taken advantage of, if the interchange of heat is to be between the waste gases and the gas liquor to be distilled.

If one discusses this question with continental sulphate of ammonia experts, it will be pointed out by them that it is wrong in principle to use gas liquor as the medium of interchange for the waste gases from the saturator. They say: "On ne peut songer à réchauffer à plus de 80 degrés les liquides à traiter, car il est nécessaire de faire traverser les appareils distillatoires d'une quantité minimum de vapeur pour produire l'entraînement de l'ammoniaque afin d'atteindre un épuisement convenable."

Although the consideration which the continental experts have in mind is appreciated, it is feared that actual practice does not substantiate their view.

Assuming the ammonia concentration in the steam-ammonia stream above the liquid of the top chamber but one of the free still is 10 per cent, the idea underlying the continental view is that by introducing the preheated gas liquor at 80° C. no dilution of the ammonia content is likely to take place, whereas by preheating the liquor to 90° C. or more, steam or other diluent will be evolved preferentially, and the ammonia concentration will be weakened. The measure of such dilution will, according to the continental viewpoint, if the author's interpretation of it is correct, represent the disability accompanying a too highly preheated gas liquor.

The author's point of view on this subject is this: It is no doubt true that dilution of the ammonia in the steam-ammonia stream does arise by feeding gas liquor preheated to 90°-100° C., as compared with 80° C., but the dilution is not attributable to the evolution of steam. Rather is this due to the immediate disengagement of hydrogen sulphide and carbon dioxide. The nearer the gas liquor attains boiling-point, on discharge to the still, the more effective will be the decarbonation and desulphurisation, on the principle that chemical combination has been disrupted, that the three principal gases—ammonia, hydrogen sulphide, and carbon dioxide—exist in solution not as ammonium hydrosulphide, ammonium carbonate, and ammonium bicarbonate, but, obeying Henry's law, as free gases in water under identical conditions. In view of the greater solubility of ammonia, as contrasted with hydrogen sulphide and carbon dioxide, the two latter gases are instantaneously released, and hence tend to cause dilution. The nearer the preheated gas liquor approaches the boiling-point the greater the dilution. But it must be remembered that in the event of partial decarbonation and desulphurisation the remaining hydrogen sulphide and carbon dioxide must be released at some point or other, and it is immaterial whether a relatively larger volume is discharged at the top of the free still, or whether the release is effected more slowly during the passage of the liquor

through the several compartments of the free section. If one is working under comparable conditions, with gas liquor preheated to 80°C ., the ammonia concentration of the steam-ammonia stream is not likely to be so high in the second chamber from the top as will be the case where the gas liquor is introduced at a higher temperature. The author therefore fails to appreciate the cogency of the continental viewpoint.

Apart from the above aspect, it is conceivable that by reducing the amount of live steam to be used at the still, due to increasing the temperature of the feed liquor to the still, say, as between 90°C - 100°C . and 80°C ., less dilution of the liquor traversing the still will occur during distillation, and to this extent the elimination of ammonia from the liquor will be aided.

The author has inspected several continental sulphate of ammonia plants of tolerably large size where preheating of the gas liquor has been effected *via* the waste liquor, but, curiously enough, in several of these works no attempt has been made to take advantage of the heat contained in the waste gases, which, it will be appreciated from the thermal balances under discussion, constitutes the major portion of the potential heat.

It is quite certain that whether the gas liquor be preheated by the spent liquor or by the waste gases, the available heat cannot be adequately taken advantage of.

It will be noticed that at the works at which the thermal determinations were made it is the practice to preheat the boiler feed water by means of the spent liquor. Reference to fig. 107 will show that 58,190 B.Th.U. are recovered in this way from an available 182,200 B.Th.U., representing an approximate recovery efficiency of 32 per cent. It is true that it should be possible to preheat the boiler feed water to at least 85°C - 90°C ., given interchange heaters of adequate size. But it will be found that taking the ammonia distillation plant as a separate process, and providing this plant with its complement of boilers, the ratio of boiler feed water required to gas liquor distilled is approximately 33 : 100.

It will be wise at this juncture to anticipate the natural question which will be asked, namely, Why are 33 parts of steam required per 100 parts of gas liquor distilled when the thermal balance sheet shows that only 26 parts per 100 are required? The answer to this question is this: Apart from the steam required for actual distillation, steam is needed in addition for the undermentioned purposes:

- (a) For the forced draught furnaces, for the combustion of breeze;
- (b) For the ejection of the sulphate of ammonia magma from the saturator, and occasionally to elevate the mother liquor to the saturator;
- (c) Steam for motive power; *i.e.* for the driving of centrifugals, elevators, conveyors, bagging elevators, drying towers, etc.;
- (d) For the pumping of:
 - (1) Boiler feed water;
 - (2) Water for cooling the waste gases, and
 - (3) For pumping gas liquor and devil liquor;

and (e) For the production of cream of lime, and the pumping of this to the stills.

When the above requirements are duly considered, it will be seen that the ratio is a reasonable one, especially so if a correct idea is formed of the extent to which labour is minimised by the adoption of mechanical handling of the final product by the provisions indicated.

On reflection it will be seen that in much the same way as the volume of gas liquor is too small for admitting of effective utilisation of the heat available in the waste gases leaving the saturator, so is the volume of boiler feed water too exiguous for effective utilisation of the available heat of the spent liquor.

To reverse the order of things, and utilise the boiler feed water as the medium of heat exchange between the waste gases, and to preheat the gas liquor *via* the spent liquor, does not assist matters; indeed, on the contrary, it rather militates against more effective utilisation.

How then can advantage be taken of the heat at one's disposal? One has to enter the domain of the evaporator engineer to find a parallel problem and its solution. Owing to the high latent heat of water, it was recognised several years ago that the evaporation of water was expensive. By employing

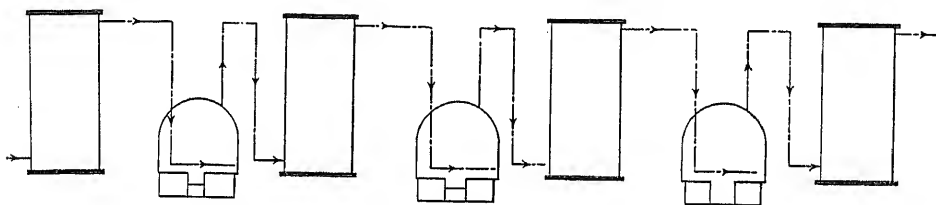


FIG. 109.—SCHEMATIC DIAGRAM OF MULTIPLE EFFECT DISTILLATION PLANT.

steam as the heating medium in another apparatus, a large portion of the latent heat is recovered, and a further evaporation is obtained. It is seen that this method of utilisation need not stop at one apparatus, but that a multiplicity of apparatus can be used. Hence the origin of the well-known system of multiple effect evaporation.

Although the disengagement of ammonia from ammoniacal solutions is not an evaporation problem, yet it is obvious that a solution of the problem under discussion is to be found in the adoption of a number of stills and saturators worked in series. The waste gases from the first saturator will enter a further still, and so on. In other words, multiple effect distillation must be resorted to. Fig. 109 shows schematically the arrangement of plant.

The idea of a multiple effect arrangement of distillation plant for sewage liquors and ammonia solutions was the subject of a discussion in 1876, and again in 1881, when Young was successful in securing a patent which provided for the desulphurisation and decarbonation of ammoniacal liquor, and for the introduction of the steam and ammonia vapours from the saturators to the stills by means of injectors; but an essential consideration which would be of material consequence in these days was not contemplated.

Multiple effect distillation had apparently only a limited application on the large scale. Little published matter can be found concerning it.

The same conception was revived in the early years of the twentieth century, at the time of the introduction of the vacuum plant for the manufacture of sulphate of ammonia, erected by Ernest Scott & Co., Ltd., of London and Glasgow. In the paper read by the late J. Ballantyne, of Hamilton, on the new process, the penultimate paragraph of his contribution is significant in this connection. It reads as follows: "The foregoing description applies to what is called a 'single effect'

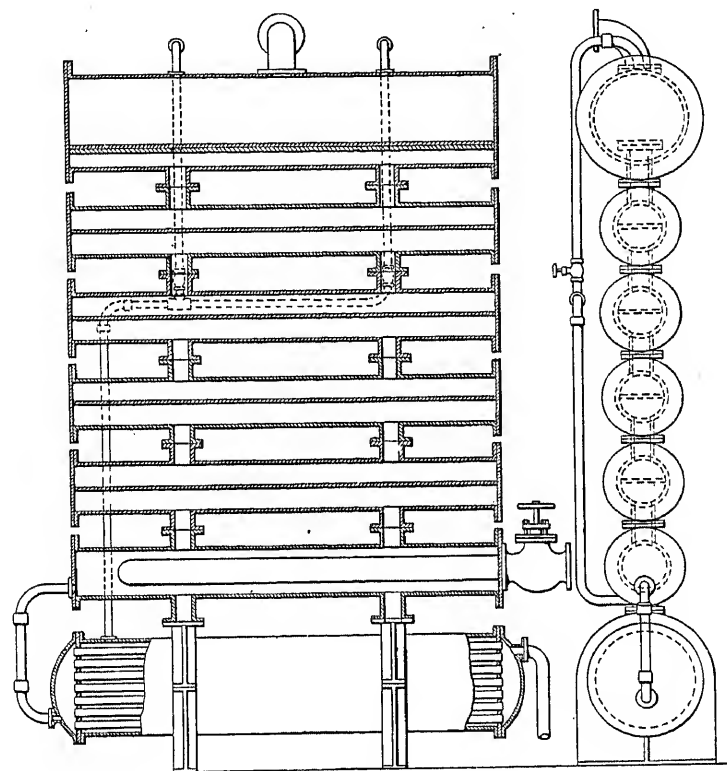


FIG. 110.—SCOTT'S AMMONIA STILL.

plant, installed at Hamilton. Although it was at first intended to erect a 'double effect' plant, it was found, however, that the former would be quite large enough for the purpose desired. Where exhaust steam may not be available, or not sufficient, and live steam has to be used, a considerable saving will be obtained by the double effect plant, as the waste gases from the first still and saturator, as shown on fig. 110, are utilised for heating the liquor in the lower part of the second still. Only one preheater, condenser, and vacuum pump are required, and as practically double the quantity of liquor can be dealt with, and therefore double the quantity of sulphate made, it must be evident that the saving in fuel per ton

of sulphate made will be much greater in the double effect plant, besides a smaller floor space being required."

Here, again, is the same idea, but, singularly enough, an essential factor is not contemplated. What that factor is it is impossible for the author to say, having regard to pending patents. It is believed, however, that it has been made clear that the existing arrangement of sulphate of ammonia plant cannot be operated with a much better steam consumption per lb. of ammonia dissociated from gas liquor of 1.59 per cent ammonia than :

$$\frac{260 \text{ lb. steam}}{15.55 \text{ lb. ammonia}} - \frac{32}{100} \text{ (heat recovered in boiler feed water)} = 11.37 \text{ lb.,}$$

whereas if the process is a self-supporting one, as has been affirmed, it is evident that some radical improvement or alteration must be made.

It should be observed that the figure of 11.37 lb. of steam per lb. of ammonia dissociated would be improved if some other means of dealing with the devil liquor were found. This noxious liquor has to be heated from a temperature of about 50° C. to the maximum still temperature, 105° C., and as there are 18 gallons, or 183.6 lb. per 15.55 lb. of ammonia, a simple calculation reveals that 1.2 lb. of steam per lb. of ammonia are used in this operation.

Summarising the position : it may be stated that so far as the existing arrangement of sulphate of ammonia plant is concerned, an improvement in the steam consumption can be effected by taking greater advantage of the potential heat of the spent liquor, although where the ammonia distillation works is more or less self-contained, full use cannot be taken of the thermal units at disposal, on account of the disparity between the relative volumes of boiler feed water and spent liquor.

Economy of steam can also be effected if a better method of dealing with the devil liquor than the distillation one is devised.

A rational solution of the problem, however, undoubtedly lies in the adoption of a multiple effect system of distillation, but the arrangement of plant will need to contemplate considerations hitherto unthought of. Any improvement in the form of the plant foreshadowed will necessarily add to its complication, but it is not imagined that any one will be inclined to support simplicity and extravagance as contrasted with something a trifle more complex, but peculiarly economical.

CHAPTER X

THE WORKING AND CONTROL OF CONTINUOUS AMMONIA STILL

HAVING ensured that the still is satisfactory in design, and is calculated to fulfil its specific duties as regards (a) the quantity of liquor to be distilled, (b) the satisfactory elimination of the ammonia with (c) a minimum steam consumption, then it is necessary to give attention to the equipment of the distillation plant in the matter of :

- (1) Constant steam supply,
- (2) Constant liquor feed, and
- (3) Constant discharge.

The vital importance of the latter factors will be obvious.

CONSTANT STEAM SUPPLY

The pressure against which the steam has to work in the fixed still, where usually 90 per cent of the total steam is admitted, is the pressure of the combined liquor seals of the several trays, both free and fixed sections, in addition to the seal of the saturator if sulphate of ammonia is being manufactured, or of the receivers (if these have a sealed distribution pipe), in the case of liquor ammonia, or concentrated ammonia liquor manufacture. Further, with sulphate of ammonia manufacturing plant there is the pressure created by the resistance of the preheaters, coolers, and pipe lines. The total pressure will obviously vary with different plant, but usually does not exceed $3\frac{1}{2}$ lb. per square inch.

As regards the steam admitted to the liming chamber; the pressure against which this will have to work may be of the order of 5 to 6 lb. per square inch. It is clear, therefore, that some form of reducing valve must be used in order to reduce the pressure at which the steam boilers normally work, to one compatible with the requirements of the still. The "Ross" reducing valve (fig. 111), as made by Bryan Donkin & Co., has proved very satisfactory in this respect. The steam at high pressure enters tangentially, and passes through the slotted holes of the cage, and the extent to which the spindle, which carries two inverted mushroom type valves,

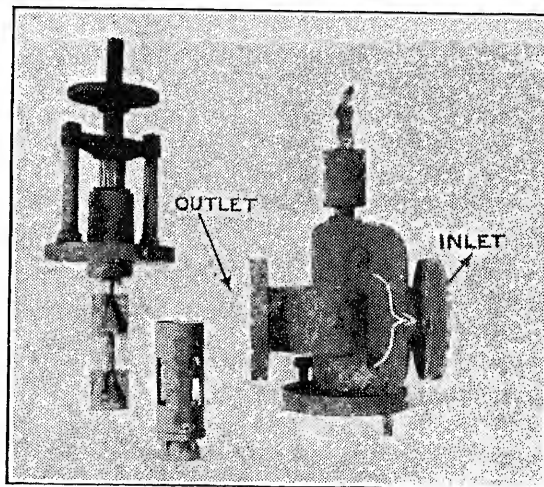


FIG. 111.—REDUCING VALVE.

is weighted, determines the size of the apertures through which the steam has to pass, and incidentally governs its pressure. Suitable drainage provision is made for any slight condensation arising.

CONSTANT LIQUOR FEED

This may consist of a balanced tank of the enclosed type, with suitable vent, or a Rotameter or a graduated sight feed arrangement, consisting of a globe, inside which is placed a pipe having a series of vertical stepped holes graduated to represent various rates of flow. Illustrations of two graduated sight feeds are given in figs. 112 and 113.

An arrangement for ensuring a satisfactory and uniform feed in connection with large units of plant is shown in fig. 114. This arrangement provides for direct pumping of the liquor to the still, the rate of feed being governed by a cock, to which is fitted a graduated quadrant. As a means of maintaining the delivery of the liquor from the pump at a constant pressure, a connection is made from the delivery line to the suction, and inserted in this line is a spring type of by-pass valve as shown in fig. 115. The principle on which this valve works is this: the liquor floats the mushroom valve against the pressure of the spring, which can be adjusted.

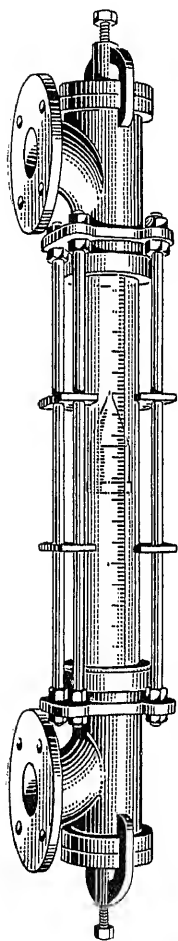


FIG. 112.
ROTAMETER.

CONSTANT DISCHARGE

This depends essentially on the maintenance of the two foregoing conditions. It has been found in practice that the setting of the outlet cock can be best adjusted by providing it with a graduated quadrant and pointer. Constant discharge float valves are provided by several firms, for answering the same purpose.

STARTING AN AMMONIA DISTILLATION PLANT

In starting a new unit it will be assumed that the engineering staff has definitely ensured that the trays of the still are dead level, otherwise a grave difficulty prejudicially affecting the efficiency and subsequent working of the plant has occurred.

It is known that unsuspected mal-alignment of the elements of stills has been the cause of trouble in their operation; the capacity of the plant being seriously affected, the waste liquor being high in ammonia content, and the steam and ammonia stream low in ammonia concentration, causing trouble in subsequent parts of the plant. Fig. 116 represents a complete distillation unit, type "A."

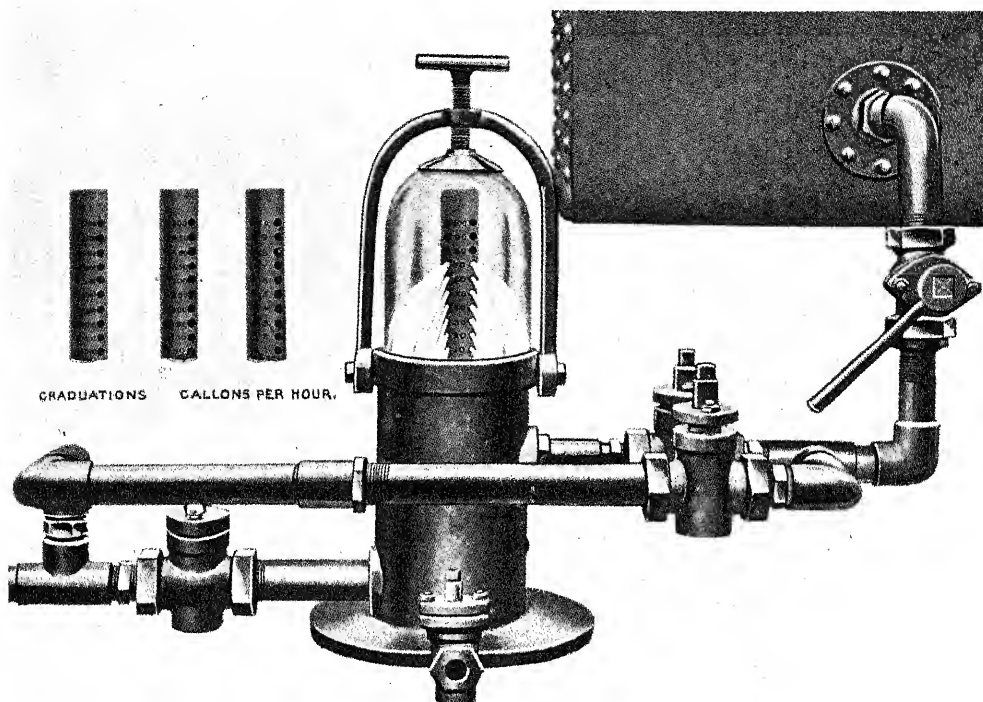


FIG. 113.—WILTON'S GRADUATED GAS LIQUOR FEED APPARATUS

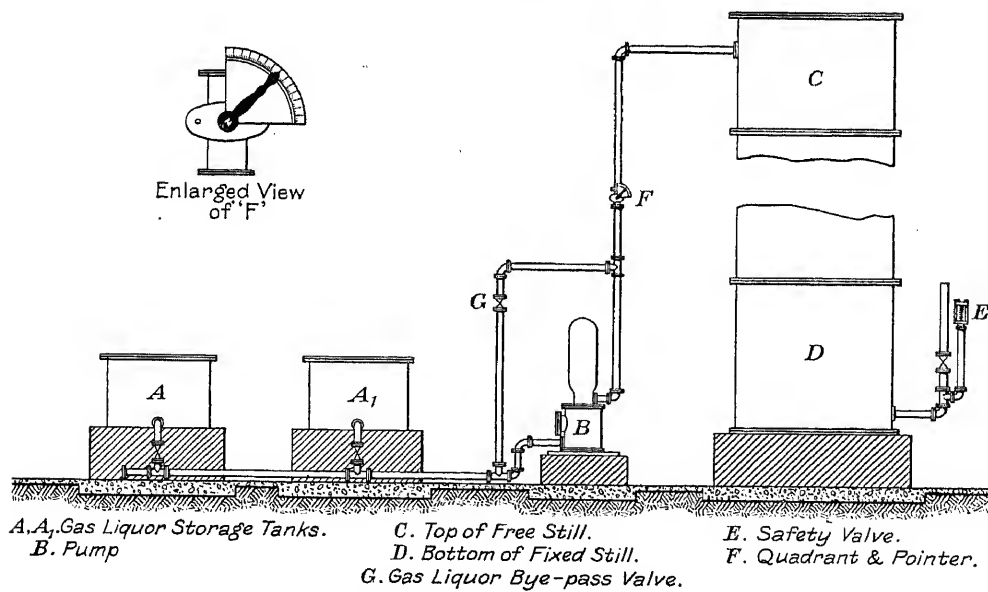


FIG. 114.—DIAGRAMMATIC SKETCH OF CONSTANT FEED ARRANGEMENT.

Prior to the admission of steam to the still for heating purposes, it is important that all the trays should be sealed, as well as the steam pipe or jets in the liming chamber and the fixed still. The heaters and coolers should also be filled with gas liquor and water respectively, so as to prevent possible fracture of the pipes when the cold liquor is first introduced. Provision should be made for venting the air from the still. A suitable arrangement is to take a $1\frac{1}{2}$ -inch wrought-iron pipe from the cast-iron pipe leading from the still to the saturator, or the dephlegmator in the case of liquor ammonia or concentrated ammonia liquor plants, and to provide this with a cock, allowing the discharged foul air to be emitted to the atmosphere.

The practice of venting a still inside the still-house is a dangerous one, and should be discountenanced.

The gradual admission of steam to the still is important, particularly with

the types of distillation apparatus which consist of a series of double-flanged pieces which are heavy in character. Such stills must be cautiously heated, otherwise the irregularities of expansion will cause fracture.

With a new still of tolerably large capacity, at least twelve hours should be allowed for raising the temperature to the requisite degree for the admission of the gas liquor.

Thermometers should be provided, preferably fixed in suitable oil pockets, on the liquor inlet to the preheaters and on the outlet to

the still, and similarly thermometers should be fixed on the waste gas inlet to the coolers, and on the outlet.

It is also desirable to arrange a centrally situated gauge board (see fig. 117), so that the boiler and reduced steam pressures, as also the pressure of the liquor being pumped to the stills and the resistance thrown by the passage of the waste gases through the heaters and coolers and subsequent pipe lines can be observed without difficulty by the operator of the plant.

It is equally important that each of the stills of the "A" type, and the columns in the case of types "B" and "C" should be provided with an efficient spring safety valve of a simple construction (see fig. 118), which can be easily adjusted and set to relieve at any predetermined pressure, and which can be finally sealed against interference.

The heating of a still after temporary stoppage need not occupy more than four hours. After the top portion of the still has become warm, the cock of the vent pipe can be closed, and the steam sent forward through the saturator (the cracker

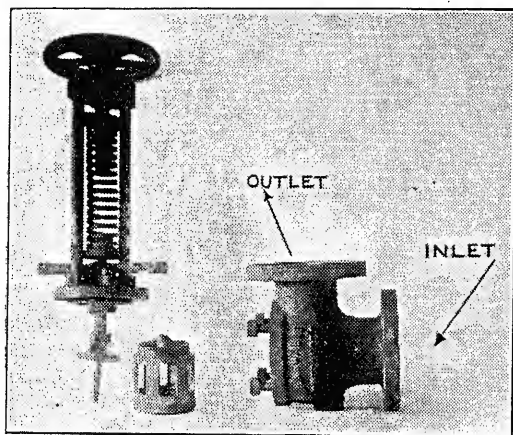


FIG. 115.—GAS LIQUOR BY-PASS VALVE.

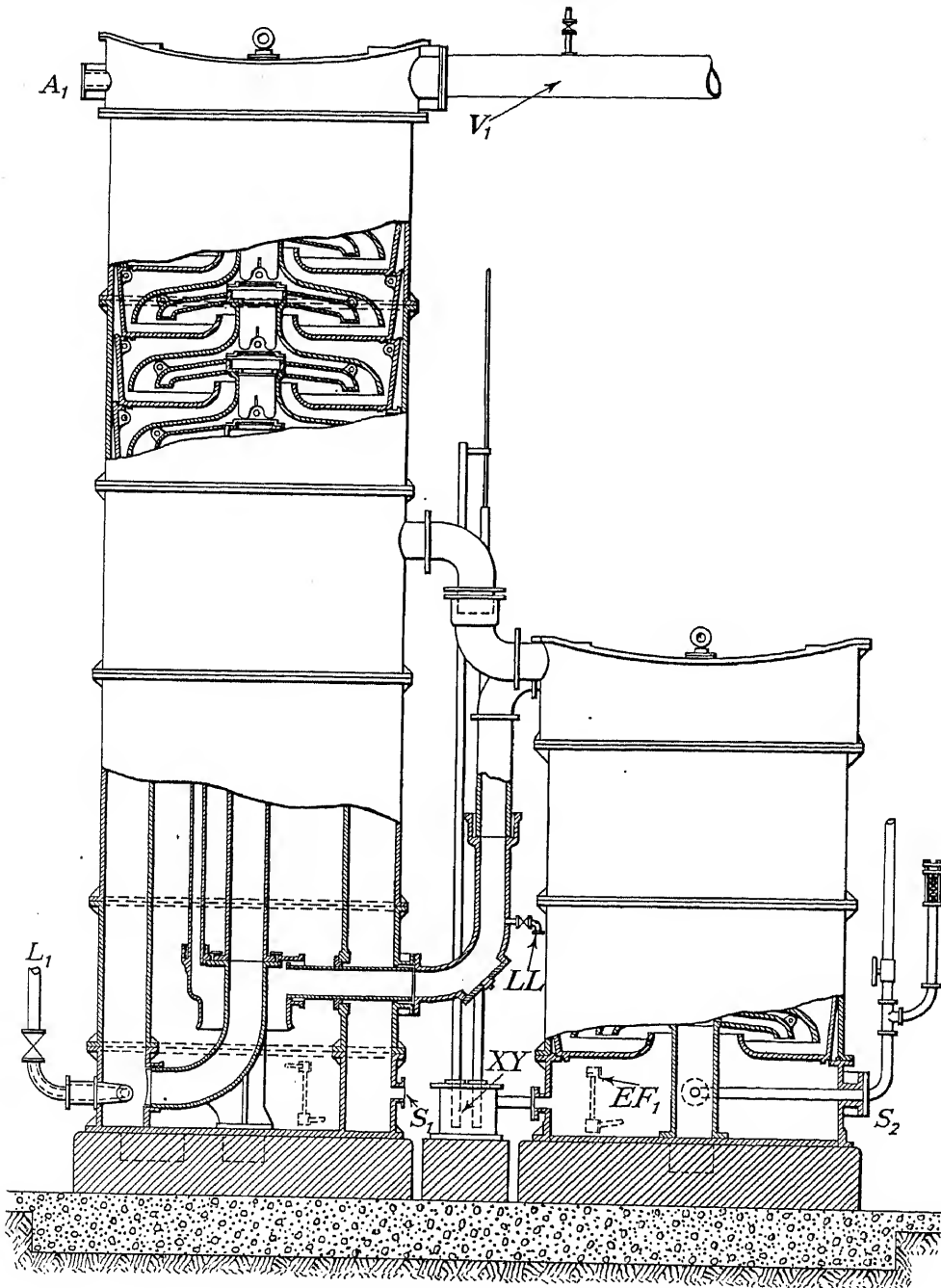


FIG. 116.—DR. CARPENTER'S STILL (SECTIONAL DRAWING).

pipes in which have been sealed with mother liquor), or to the dephlegmators, etc. in the case of liquor ammonia or concentrated ammonia liquor manufacture. It is important, in sulphate of ammonia manufacture, that the devil liquor outlet cocks

should be opened immediately the heaters become warm, in order to allow of the automatic withdrawal of the condensation which must arise, due to the cooling influence of the gas liquor and water with which the heaters and coolers are filled.

Prior to feeding the still with gas liquor it is wise to pump, or gravitate, milk or cream of lime to the still, so that by the time the gas liquor is introduced, there is a complement of boiling milk or cream of lime in the liming chamber.

Reference to the drawing (fig. 116) will show that steam must be

admitted to S_1 and S_2 , that the liquor feed is at A_1 , that the lime feed is at L_1 , that the steam-ammonia stream issues at V_1 , that samples of the limed liquor can be drawn at LL , and that samples of effluent liquor can be drawn at EF_1 .

The raising of the temperature of the apparatus to this point is usually done with the reducing valve full open, but immediately the liquor feed is commenced the reducing valve is brought into operation by removing one or more of the weights on the spindle. The next step is to direct attention to the passage of the limed liquor to the fixed still, the connecting pipe to which is usually provided with a cock (LL), so that samples can be drawn from time to time. As soon as the limed liquor enters the fixed still a further and final adjustment of the steam reducing valve and the steam inlet valves to the stills is necessary.

Temperatures at the essential parts of the plant should be taken at regular intervals, and should be recorded. Equally, the liquor feed gauge board should be watched very carefully so as to make certain that a uniform feed is being obtained. Records of the liquor fed to the still should be made half-hourly.

It is usual to connect a cast-iron pot XY to the fixed still, and to provide such

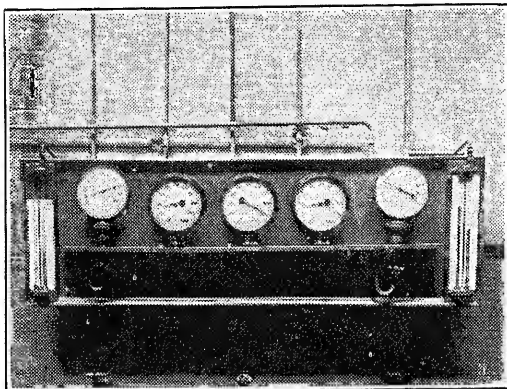


FIG. 117.—GAUGE BOARD.

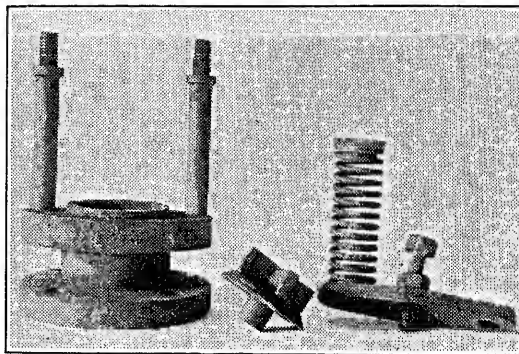


FIG. 118.—SAFETY VALVE.

pot with two vertical pipes, one of which is carried almost to the foot of the pot and which is provided above the pot with a glass tube suitably connected to give an indication of the working level of the still, the other pipe being similarly equipped (but not provided with a glass tube) in order to serve as a safety-valve.

One other point of importance as affecting the operation of gas liquor stills has reference to the necessity for a uniform feed of devil liquor along with the gas liquor.

FACTORS GOVERNING EFFICIENCY

As is indicated in the introductory chapter, the primary factors contributing to efficiency are (a) comparatively high productive capacity, (b) maintenance of a satisfactory waste liquor, say, not exceeding 0.01 to 0.015 per cent NH_3 , and (c) minimum steam consumption per unit volume of liquor distilled.

COMPARATIVELY HIGH PRODUCTIVE CAPACITY

This affects labour; the higher the productive capacity the less the cost of labour. The rate of working depends on the design of the still, the composition of the liquor as regards free and fixed ammonia, and the efficiency of the liquor preheating arrangements.

It will be seen from the thermal balance sheet furnished in the previous chapter that the plant on which the thermal determinations were made was operating very satisfactorily in this connection.

MINIMUM STEAM CONSUMPTION

As is pointed out in the foregoing chapter, there is a larger volume of heat

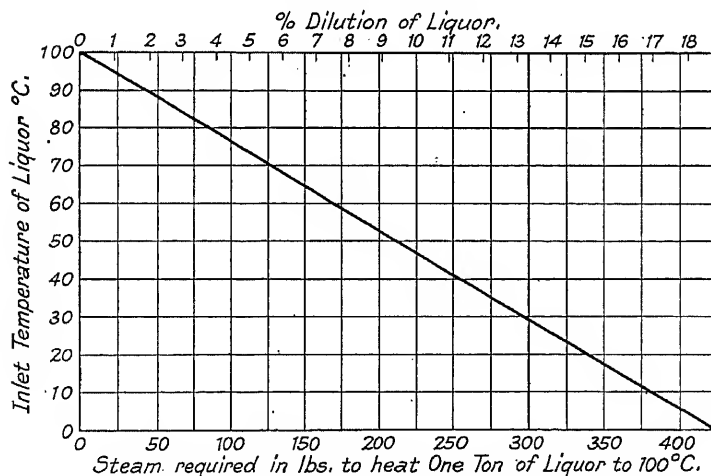


FIG. 119.—GRAPH SHOWING STEAM REQUIRED TO HEAT AMMONIACAL LIQUOR.

available for preheating the liquor than can be taken advantage of. It is therefore regrettable to find many works throughout the country only partially preheating

their feed liquor, say, to 50° or 60° C. (not more). Fig. 119 serves as a striking commentary in such cases. For example, at a works only preheating its liquor to 60° C., it is seen that approximately an extra 135 lb. of steam are required per ton of liquor, as contrasted with a works preheating the liquor to 90° C. Moreover, the dilution of the liquor is increased by at least 7½ per cent, and this must influence the efficiency of the removal of the last traces of ammonia.

IDEAL CONDITIONS

Ideal conditions represent (a) the distillation of the maximum quantity of preheated liquor with (b) a minimum steam consumption on the fixed and free stills, and (c) the liberation of all the CO₂ and H₂S from the liquor prior to its reaching the liming chamber. (d) The feed of cream of lime should be regular, and as strong as possible, and (e) the spent liquor should be practically denuded of ammonia (both fixed and free), and should not contain an undue excess of caustic lime, or have suffered undue dilution in its passage through the still. The importance of several of the above points has been urged in previous chapters, and it is therefore unnecessary to labour their significance.

The attainment of the ideal conditions outlined on the actual plant involves intelligent process workers, combined with efficient control. The latter may not always be available or possible, on small works.

Tests at various parts of the plant will soon reveal delinquencies, or otherwise indicate that the plant is operating satisfactorily. It is desirable that the interpretation of any such tests should be correct, and well appreciated. Tests, after all, are useless except it is realised what they imply, and the necessary steps are taken not only to rectify any defects, but to prevent their recurrence.

CONTROL AND OPERATION OF THE STILL

The importance of constant steam and preheated gas liquor supplies has already been urged. In addition to these, attention should be directed to maximum preheating of the gas liquor, and the maintenance of a constant temperature. The strength and temperature of the cream of lime should also be carefully watched. Cold cream of lime unnecessarily chills the still, reducing the capacity. A familiar rattling noise in the liming chamber indicates an insufficiency of steam. Too much steam in the liming chamber is undesirable. A weak solution of lime obviously causes dilution, and must be obviated.

The temperatures and pressures at various parts of the still should be recorded, as also any adjustments which are made. In this way, valuable data are amassed, which cannot be but serviceable in indicating the condition and cleanliness of the still, and its accessory parts.

A continuous sample of the waste liquor (an apparatus for which is shown in fig. 120) should be taken, and a definite standard adhered to in this connection. Experience suggests that 0.015 per cent NH₃ in the waste liquor is an economic

strength, as working below this limit simply resolves itself into the recovery of an exiguous amount of ammonia at the expense of labour in the operation of the plant, when manufacturing sulphate of ammonia. The devil liquor should be tested periodically from the points of view of its freedom or otherwise from ammonia and its volume. The presence of free ammonia only, indicates that ammonia is passing the saturators. When both free and fixed ammonia are present in the devil liquor, then the inference to be drawn is that the tubes of the heaters are leaking, and this defect should be arrested at the very outset. An unduly large volume of devil liquor indicates a high consumption of steam in the distillation of the liquor, or otherwise the leakage of water in the coolers.

Early indications are furnished of the fouling of the stills by the necessity for reducing the volume of steam being introduced thereto, and it will be found, other things being equal, that the effluent liquor gradually increases in point of ammonia content. If tar accidentally becomes mixed with the liquor, in order to prevent this

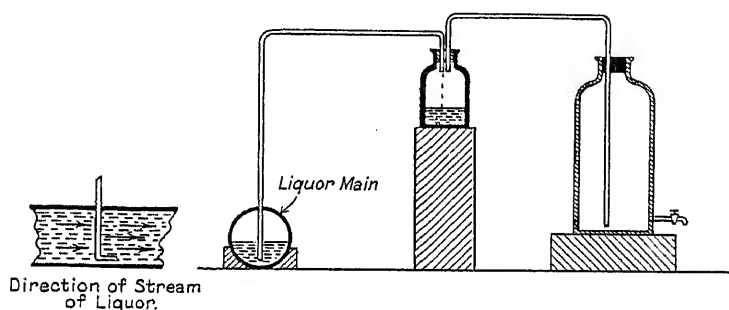


FIG. 120.—SAMPLING DEVICE.

adversely affecting the working of the still, the preheaters and pipe lines to the still should be discharged, such discharged liquor being returned to a tank for separation or other suitable treatment.

TESTS

Hereunder will be found, in succinct form, the tests which should be applied to determine the efficiency of the working of the plant.

- (1) *Free Ammonia in Effluent Liquor*.—100 c.c. of effluent liquor are distilled in the usual way into standard sulphuric acid, and the excess of acid titrated with standard caustic soda, using methyl-orange, as an indicator.
- (2) *Fixed Ammonia in Effluent Liquor*.—To the cooled residue from (1) there is added an excess of 10 per cent caustic soda solution, and the mixture distilled into an excess of standard acid, and titrated with standard caustic soda, as before.
- (3) *Excess of Caustic Lime in Effluent Liquor*.—100 c.c. of a representative sample of well-mixed effluent liquor containing all the solid matter as it leaves the fixed still, are placed in an ammonia flask, and boiled until the free ammonia

has been driven off, and to the cooled residue is added 20 c.c. of a 10 per cent sulphate of ammonia solution. The mixture is then distilled into standard acid. Any free lime present liberates ammonia from the sulphate. After the distillation the acid is titrated with standard caustic soda. Each 1 c.c.

of $\frac{N}{I}$ acid neutralised is equivalent to 0.028 gram of caustic lime (CaO);

any excess of this material can be readily calculated.

- (4) *CO₂ in Liquor from Free Still.*—Take 100 c.c. of a filtered sample, and transfer to a beaker; add 10 c.c. of a 10 per cent CaCl₂ solution, heat to boiling, and filter through a small paper. Wash with boiling water until the washings give no precipitate with silver nitrate (acidified with nitric acid). Transfer the filter paper to a beaker, and add excess of standard hydrochloric acid, well stirring to dissolve all the CaCO₃ from the paper, then titrate with standard sodium carbonate, using methyl-orange, as an indicator. Each c.c. of acid neutralised by the precipitated CaCO₃ represents 0.022 gram CO₂ per 100 c.c., which would require lime equivalent to 0.028 gram, or about 0.28 lb. per 1000 gallons of liquor used.

The results of all such tests as the above should be recorded in suitable form, along with data bearing upon (a) the free and fixed ammonia content of the ammoniacal liquor used, (b) the strength of the cream of lime used, (c) steam pressure, and (d) position of valves and readings of thermometers and pressure gauges, etc., when best conditions of working have been attained.

With such data in one's possession, adjustment of conditions to ensure the most efficient and economical working should not be very difficult.

INTERPRETATION OF RESULTS

Having secured the best working conditions, then it is only necessary to maintain the plant to this standard in all respects. It will be appreciated that ideal conditions cannot be maintained uninterruptedly, as occasional variations will arise, even in the best-regulated works.

Results will vary, as is indicated below:

- (1) As regards free ammonia in the effluent liquor:

- (a) If the standard figure of 0.015 per cent is adhered to, the still has operated well, even though there may have been fluctuations in the steam and lime supplies.
- (b) Should a better figure than the standard one be secured, then there has been too much steam used.
- (c) If the standard is exceeded, there has been an inadequate supply of steam at S_1 , or otherwise an irregular supply of cream of lime.

- (2) As regards fixed ammonia in the effluent liquor:

- (a) If the standard figure has been reached, this implies efficient working.

- (b) If the standard figure has not been reached, there has been an insufficient supply of cream of lime.
- (3) As regards CO_2 content in the effluent liquor leaving the free still :
 - (a) If the standard figure has been reached, this denotes good working.
 - (b) If the standard figure has been exceeded, there has been an inadequate supply of steam to the free section of the still.

If these points are carefully watched, and daily records are kept and plotted, and brought prominently to the notice of all concerned, efficient and economical working should be the inevitable outcome.

SEAL POTS

To prevent the escape of steam, etc. from the foot of the fixed still or fixed

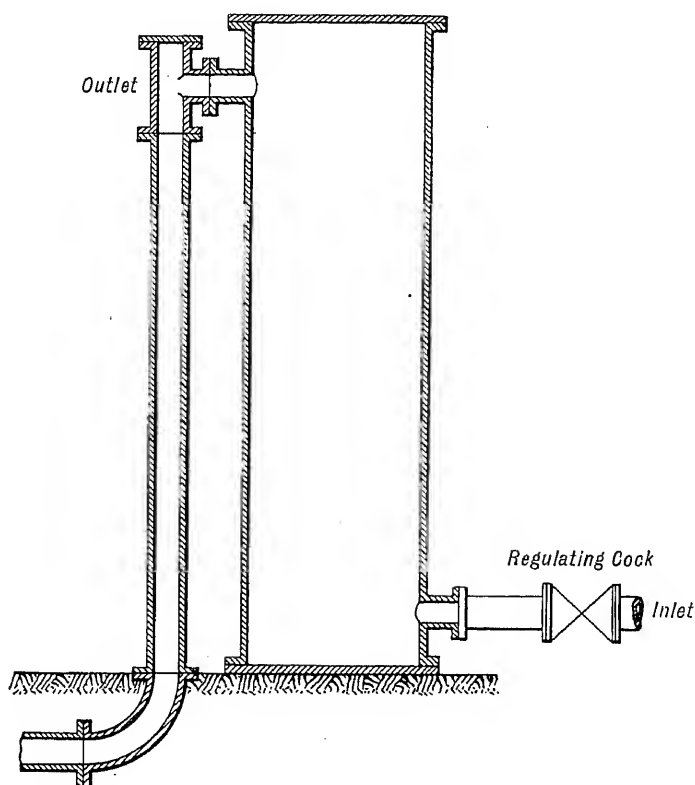


FIG. 121.—SEAL POTS (a).

segment (the last chamber of which should be provided with a gauge glass so that the depth of the liquor can easily be seen by the operator), it is necessary to provide some

suitable seal pot, or equivalent arrangement, in order to ensure that the steam admitted to the fixed still will proceed through the several chambers, and not by-pass

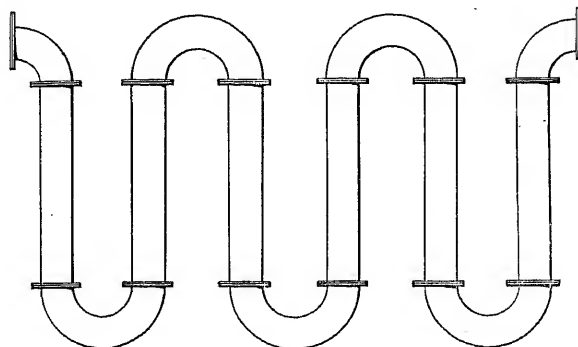


FIG. 122.—SEAL POTS (b).

the apparatus. There are several kinds of seal pots, as is shown in figs. 121, 122, and 123. Some seal pots usually take the form of a double-flanged pipe with blank

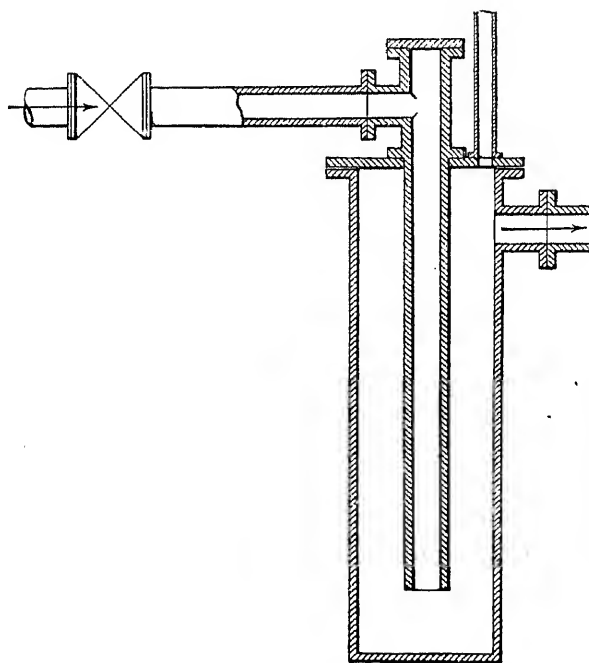


FIG. 123.—SEAL POTS (c).

plates top and bottom. The waste liquor pipe is provided with a regulating cock immediately on the outlet from the fixed still, and this pipe is led in some cases to

the foot of the seal pot, which is provided with an outlet near the top ; or the waste liquor pipe is fixed inside the seal pot, discharging the waste liquor near the foot ; the outlet from the pot being near the top. The former type is placed on the ground

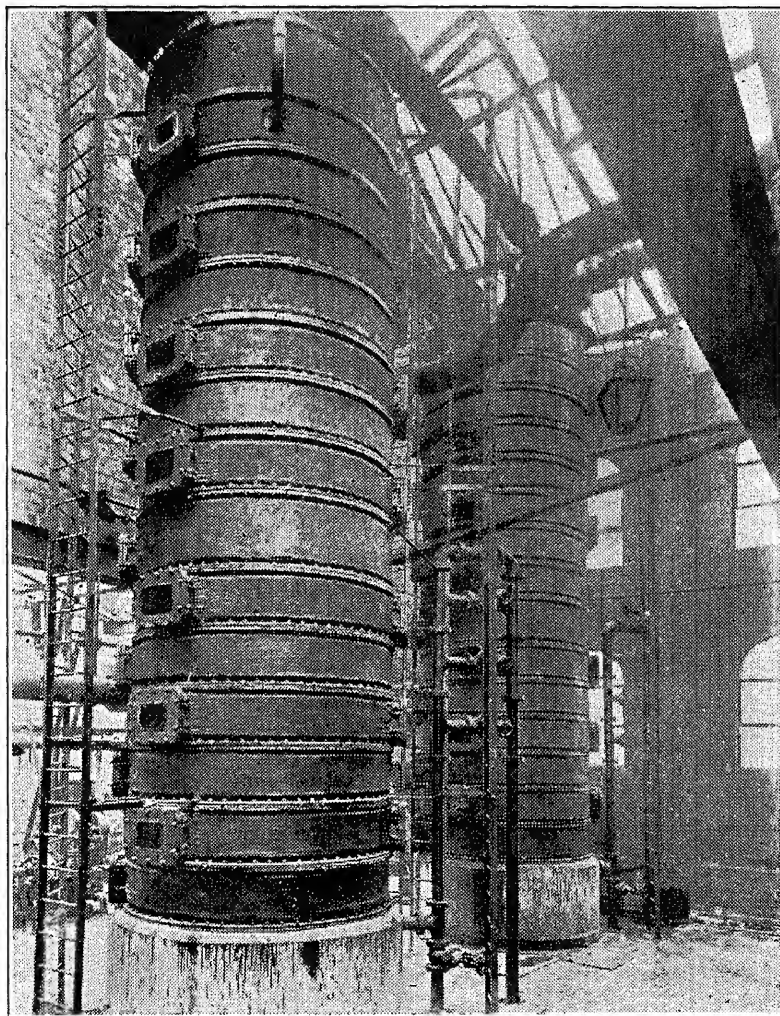


FIG. 124.—ADJUSTABLE SEAL ARRANGEMENT (GAS LIQUOR STILLs).

level ; the latter type is sunk into the ground. It is preferable that a vent pipe should be provided in connection with the seal pot, so as to prevent steam "locks." An adjustable type of sealing device is shown in fig. 124.

DEVIL LIQUOR STORAGE TANK

In the case of sulphate of ammonia plants where the waste gases from the saturator are passed to the gas liquor preheaters and coolers, the condensed vapours resulting from the cooling influence of the ammoniacal liquor and water are known as "devil liquor," by reason of the evil-smelling and noxious character of the latter. It is important that proper steps should be taken in a well-organised works for the collection and treatment of this liquor. The liquor is run to a sectional plated cast-iron tank, suitably lagged, which will allow of five to six hours' accumulation. Opinions vary as to the best method of treatment. At many works the devil liquor is pumped along with the gas liquor, and the mixed liquors are distilled. The only disadvantage is that expense is entailed in the matter of steam, and many of the impurities of the gas liquor which are arrested with the devil liquor, such as pyridine, creosote oil, naphthalene, etc., are again returned to the saturator. Obviously, for the production of white salt, it is essential that these impurities should be disposed of in another way, and not recirculated to the saturator.

Moreover, it must be remembered that the distillation of devil liquor represents approximately a 20 per cent reduction of the capacity of the still, which is another important consideration.

In the cases of concentrated ammonia liquor and liquor ammonia plants, the vapours are usually condensed in a scrubber which is provided for arresting the small percentage of ammonia which accompanies the H_2S , CO_2 , and other vapours leaving the dissociator.

STOPPING THE STILL

In the case of stills attached to a sulphate of ammonia plant, it is necessary to remove the salt from the saturator, and to work down the charge therein as low as possible, subsequently adding a quantity of acid, before taking the necessary steps to render this piece of apparatus inactive.

Having discontinued the liquor supply to the still, it is advisable to maintain the feed of cream of lime for about fifteen minutes. The steam supply should be continued for about half an hour after the liquor feed has ceased. In this way it is possible to ensure the removal of the last traces of ammonia from the gas liquor remaining in the still. Prior to turning off the steam, the vent in the still outlet pipe should be opened, so as to obviate any syphoning of the contents of the saturator into the still or pipe lines, due to the cooling of the still. In the cases of concentrated ammonia liquor, or liquor ammonia plants, all the latter instructions apply except those having reference to the sulphate saturator.

BLOCKAGES AND CLEANING : PRACTICAL NOTES

It has already been indicated that early indications of the fouling of the stills will reveal themselves.

If the liquor contains tar, even the merest traces, it will be appreciated that

light hydrocarbons will be distilled off, and pitch will remain and collect under the overflows. Eventually these will become completely obstructed, thus causing the ammonia liquor to descend through the gas liquor pipes.

Tar and sedimentary matter are the only obstacles likely to affect the working of the top section of the free still. The seal or dip pipes conveying the fixed ammonia to the bottom of the liming chamber are in many cases too large in diameter. Obviously, a reasonable velocity is required to prevent the accumulation of lime salts around the foot of the pipe, with the possibility of blockage. The periodical flushing-out of the liming chambers, say, every twelve hours, or every day, is an advantage. The arrangement shown in fig. 106 answers satisfactorily. At some works the drain cock on the liming chamber is opened once every day to flush-out any sediment which may have deposited. The defect in this arrangement is the possibility of the drain cock becoming obstructed. It will be found that the foregoing arrangement of flushing the liming chamber has much to recommend it.

The progressive increase of ammonia content in the waste liquor when all other governing factors are normal, and the frequent rising of the liquor to the top of the gauge glass of the liming chamber, are unmistakable signs of the accumulation of lime deposits in the fixed section. Cleaning is necessary if the efficiency of the still is to be regained. Even where manholes are provided on a still with separate fixed ammonia column, it is generally the quickest and surest way to dismantle the sections. Should the fixed ammonia section and liming chamber frequently become blocked, it can safely be inferred that the decarbonation of the liquor in the free section is not satisfactory. The tests already indicated should at an early period disclose these imperfections. It is imperative that the formation of calcium carbonate should be avoided if frequent cleaning is to be obviated. Occasionally dirty milk of lime may cause trouble of the latter order.

Copper and brass are readily attacked by ammonia. The steam valves used in connection with an ammonia still should be of the all-iron type, as also should the gauges and cocks, etc.

It is advisable to have the stills insured against accident in the same way as steam boilers. This ensures an independent examination by a boiler inspector, and the report of such inspector cannot be but helpful. Explosions in connection with ammonia stills are not unknown.

Assuming an obstruction has been located, and the exigencies of the plant demand an immediate stoppage of the still for cleaning, it is scarcely necessary to issue a caution against opening a hot still, not only on account of the ammoniacal vapours, but by reason of the possibilities of scalding. If circumstances demand the immediate opening up of a still, then the best procedure is to cool down with tepid water at first, and finally cold water afterwards, a supply of which should always be coupled direct to the stills.

CHAPTER XI

THE WORKING AND CONTROL OF CONCENTRATED GAS LIQUOR STILLS AND PLANT

For several years now many small works have been faced with a real difficulty in the matter of the disposal of their production of gas liquor. The market value of the liquor has been so low that railway carriage has more than absorbed the revenue derived from its sale. In some cases it has been decided to run the liquor into the sewers, etc., rather than rail it to the chemical works.

In several of these cases grave risks have been run in discharging the liquor to the sewers. The question of nuisance or pollution has often arisen.

FINANCIAL CONSIDERATIONS

In order to assist gas managers of small works to a decision in this matter, a hypothetical case will be considered, where a small gas works carbonising 2500 tons of coal per annum, and producing 100,000 gallons of gas liquor of 8-oz. strength (1.73 per cent ammonia content) per annum, has hitherto been selling its liquor to a chemical works situated about 50 miles away.

Under the old railway rates classification, gas liquor in owner's tank wagons, 8-ton lots, was conveyed as Class A traffic. The latter rates vary with different companies, and, in some special cases, different sections of the line are the subject of special rates.

In general, the railway rate can be calculated in the following manner. It will be assumed that the tank wagon conveying the liquor is run on the Southern Railway (South-Eastern & Chatham Section).

BASIS OF RATE

Class A traffic, first 20 miles, 1.5d. per ton ; next 30 miles, 0.95d. per ton ; terminals, either end, 3d. per ton.

TABLE 41

	s.	d.
20 miles at 1.5d.	2	6
30 miles at 0.95d.	2	4½
Terminals, one end only	0	3
	<hr/>	<hr/>
	5	1½
100 per cent advance, owing to circumstances arising out of the war, plus 6d., with a maximum of 4s. 0d.	4	0
	<hr/>	<hr/>
Railway rate, per ton	9	1½
	<hr/>	<hr/>

Assuming the gas liquor is to be used in the manufacture of ammonium sulphate, and viewing the matter from the chemical works point of view, it is reasonable to take the nett selling price (March 1924) for sulphate of ammonia, 25.5 per cent, at £14 per ton. The total costs of manufacture, plus profit, may be taken as £8 per ton, irrespective of the cost of the gas liquor. Thus, a sum of £6 is available for appropriation in respect of the liquor used in the manufacture of one ton of sulphate of ammonia. It will be seen from Table 11 in Chapter III. that 14.74 tons of liquor are required per ton of sulphate of ammonia made, basing on a 100 per cent conversion efficiency. Taking a 95 per cent efficiency as a reasonable figure, $\frac{14.74 \times 100}{95} = 15.51$ tons of gas liquor are required in the manufacture of one ton of sulphate of ammonia, 25.5 per cent NH_3 .

The chemical manufacturer, therefore, can afford to pay $\frac{120\text{s.}}{15.51 \text{ tons}} =$ approximately 7s. 7½d. per ton of gas liquor, on the assumption that the gas works is responsible for the carriage of the liquor. It will be seen that the loss to the gas works represents 1s. 5½d. per ton of gas liquor. It is clear that it would be better for the gas works to run the liquor to the sewer, if this is permissible, rather than rail it.

In many cases this is not permissible, and hence the question of the economics of the production of concentrated gas liquor have to be carefully considered.

The questions to be answered are (a) what will it cost to produce concentrated gas liquor of 15 per cent NH_3 content, and (b) what saving will be effected in railway carriage?

Pursuing the hypothetical case in question: A plant to produce 2 tons of concentrated gas liquor per twenty-four hours should not cost more than £650 complete.

The approximate cost of manufacture, basing on concentrated gas liquor of 15 per cent NH_3 content, will be as shown in Table 42.

Examination of the figures reveals that the saving to be effected in the matter of railway carriage over and above the costs of concentration represents

$$13\text{s. } 10\text{d.}, \text{ or (say) } \frac{166\text{d.}}{9.13 \text{ tons}} = 1\text{s. } 6\frac{1}{2}\text{d. per ton.}$$

Therefore, instead of incurring a loss of 1s. 5½d. per ton of gas liquor in sending the liquor to the chemical works in question, if concentrated gas liquor is produced, a pecuniary saving of 2s. 11.85d. per ton of gas liquor results.

Having indicated a general basis for calculation, it will be an easy matter for any one interested to substitute figures to cover their own particular case and special circumstances.

WORKING AND CONTROL OF PLANT

As will be seen from fig. 125, the arrangement of plant for the concentration of gas liquor consists essentially of a still, preheater, condenser, and liming apparatus.

Prior to starting the plant it is necessary to seal with water the trays of the free

THE DESIGN AND WORKING OF AMMONIA STILLS

TABLE 42

	Per ton.
Steam for pumping and distillation (assuming full heat conservation), say, 20 per cent on the gas liquor distilled :	
9.13 tons gas liquor : 1.826 tons steam @ 5s. per ton for the steam	0 9 1½
Labour—three men per day @ 12s. each = 36s. for two tons	0 18 0
Interest on Capital and Depreciation, 10 per cent on £650 = $\frac{£650}{*51.4 \text{ tons}}$	1 5 9
Contingent Expenses	0 5 0
Lime	0 2 6
Cost of producing 1 ton of concentrated gas liquor—15 per cent NH_3	£3 0 4½
Coming to question (b) :	
Instead of being involved in carriage on 9.13 tons at 9s. 1½d, representing	£4 3 4
Only one ton of concentrated liquor is required to be conveyed, representing	0 9 1½
Saving effected	£3 14 2½

* 100,000 gallons = approximately 446 tons \times 1.73 per cent NH_3 = 7.715 tons ammonia.

9.13 tons gas liquor, based on 95 per cent conversion = 1 ton of concentrated gas liquor, or 1.50 ton ammonia.

Therefore, 7.715 tons of ammonia \div 0.15 ton = *51.4 tons of concentrated gas liquor (15 per cent NH_3) will be produced per year.

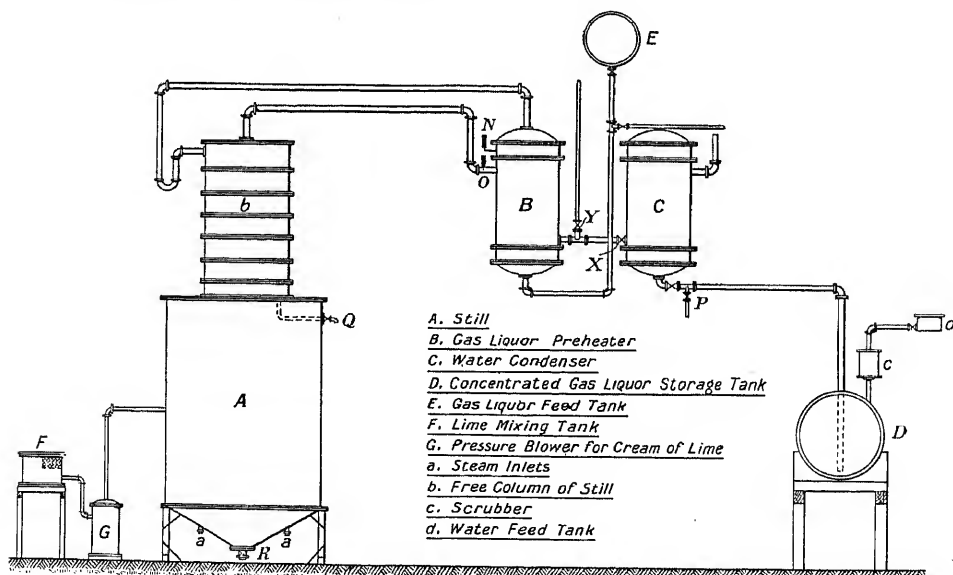


FIG. 125.—CONCENTRATED GAS LIQUOR STILL.

portion of the still and the conical bottom of the fixed still. The preheater should also be filled with water.

The valve X giving access to the vapours of the condenser should be closed and vent Y opened for the discharge of any steam or vapour resulting.

Steam should be admitted to the still at *a, a*, and the heating process should proceed slowly at the start so as to prevent any fracture of the cast-iron sections of the free still.

After the temperature has been raised to 50° C., as indicated by the thermometer N, the steam can be increased, and as soon as the temperature of the water at O reaches 90° C., then valve X should be opened and the vent cock Y closed. Meanwhile the gas liquor and cream of lime feeds should be started.

When the still operator has ensured that the feeds of gas liquor, cream of lime, and steam are regular, the next step is to give attention to the strength of the concentrated gas liquor being produced, samples of which can readily be drawn from the sampling cock P shown. The ammonia content of the concentrated gas liquor is a function of the strength of the ammoniacal liquor fed to the still and the steam consumption.

It is inexpedient to exceed 15 per cent of ammonia in the condensate, otherwise trouble may arise by reason of the crystallisation of ammonium bicarbonate and/or ammonium carbamate. If the condensate exceeds 15 per cent of ammonia, then this can be suitably diluted.

Having regard to the vapour pressure of concentrated gas liquor of 15 per cent ammonia content, care should be taken to run the condensate into the storage tank through a pipe leading to within a few inches of the bottom. In this way agitation is avoided. The vented air should also be scrubbed through a suitably packed small scrubber maintained at all times with a wetted surface. This is necessary to obviate loss of gaseous ammonia.

After the operator has adjusted the conditions of the plant so as to ensure a condensate not exceeding 15 per cent of ammonia, then it is necessary to collect at Q samples of the liquor entering the fixed still, as also of the spent liquor leaving the still at R. These samples should be submitted to test.

Given efficient working, there should be practically no free ammonia or carbonate in the former sample. If the percentage of free ammonia exceeds (say) 0.10 and carbonate is also present, then the feed of liquor should be checked or otherwise the steam increased. The sample drawn at R should be tested for caustic lime and ammonia. If caustic lime is absent, then it is necessary to increase the lime feed slightly in excess of the equivalent of the fixed ammonia which the spent liquor contains.

If caustic lime is present and the spent liquor contains more than 0.015 per cent of ammonia, then the quantity of liquor should be reduced slightly.

It is important to work with a safety valve fixed on the steam supply to the still, so that in the event of a blockage in any portion of the condensing system or pipes connected with it a suitable emergency relief is afforded. It should be pointed out that in the cooler portions of the plant, viz. the water condenser and pipes leading

to the storage boiler, it is wise to have a $\frac{1}{2}$ -in. steam pipe fixed so that the temperature can be raised at will in the event of crystallisation being suspected.

STOPPING THE PLANT

The first step is to shut off the feed of ammoniacal liquor to the still. The pumping of the cream of lime to the still should be continued for about fifteen minutes after the discontinuance of the supply of liquor.

The steam should be maintained on both the free and fixed sections of the still for half an hour after the liquor supply has been shut off. In this way the removal of the last traces of ammonia is ensured.

Immediately prior to shutting off the steam from the still, vent cock Y should be opened, in order to obviate the syphoning of the concentrated gas liquor from the receiver, which would otherwise occur by reason of the cooling of the still.

At many plants where the design of the still may cause anxiety through blockages due to accumulation of lime sludge it is usual to pump water through the still for flushing and cleansing purposes.

CAPACITY OF PLANT

The capacity of the plant shown is approximately 6000 gallons of liquor per twenty-four hours.

WORKING RESULTS

Working results of a plant similar in character to that illustrated in fig. 125, as given by D. D. Barnum (*Journal of Gas Lighting*, vol. 89, p. 841), are given below :

6100 gallons of liquor with 2.50 per cent ammonia, w/v, containing 40 per cent fixed ammonia were distilled, giving the undermentioned results :

- (a) The lime used (293 lb.) = 143 gallons of cream of lime.
- (b) The steam used (20 lb. per square inch pressure at the still) = 13,056 lb., or 10.39 lb. per lb. of ammonia.

The original 6100 gallons of liquor (2.50 per cent ammonia) yielded 856 gallons of 15.29 per cent w/v concentrated gas liquor.

EXAMINATION OF RESULTS

An examination of the above results shows that the gas liquor contained 1525 lb. of ammonia, and that the concentrated gas liquor produced contained 1307 lb. of ammonia, representing a loss of 218 lb., or 14.2 per cent. This certainly does not indicate efficient working. It is the author's experience that with satisfactory control a 97 per cent working efficiency is possible.

The principal loss in the above case would appear to have arisen from an inadequate use of lime, but the figures from one or two points of view will not bear a too rigid scrutiny.

Turning to the data having reference to the use of steam, a figure of 10.39 lb.

is given per lb. of ammonia, with gas liquor of 2.5 per cent NH_3 . Basing on calculations similar to those referred to in Chapter VI., the following figures are arrived at :

TABLE 43

	B.Th.U.	Steam.	Percentage.
1 lb. of ammonia requires for dissociation	2044	lb. 2.12	14.18
To heat 40 lb. of liquor from 70° to 220° F.	6000	6.28	42.01
To heat 1.98 lb.* of water with the lime .	300	0.31	2.07
Heat radiated in one minute	620	0.70	4.68
Lb. of water in concentrated gas liquor .	..	5.54	37.06
Total steam required per lb. of ammonia	..	14.95	100.00

* Only 293 lb. of lime were used in relation to 1307 lb. of ammonia recovered. The above is based on a 10 per cent solution of milk of lime.

Barnum's figure, if correct, is very satisfactory. It will be appreciated from what has already been said that the figure given representing the amount of heat required in the dissociation of 1 lb. of ammonia from gas liquor of varying ammonia concentrations is somewhat doubtful. Full reference has been made to this aspect in Chapter VI.

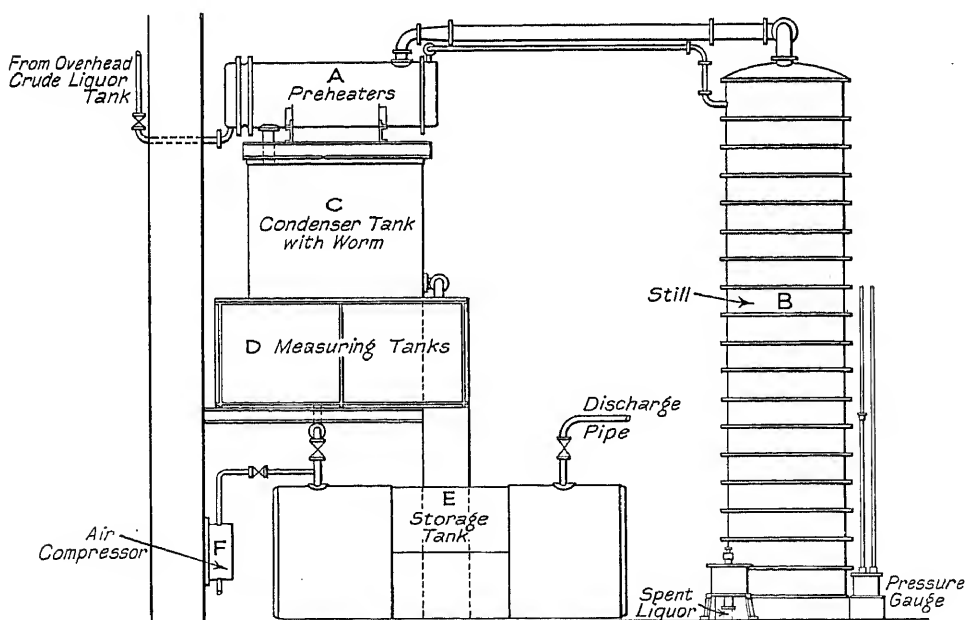


FIG. 126.—CONCENTRATED GAS LIQUOR PLANT.

Fig. 126 represents a concentrated gas liquor plant, as erected at the Paisley

Corporation Gas Works, which was described by A. S. Nisbet in a paper he read to the North British Association of Gas Managers on September 6, 1923. The concentrated gas liquor plant, as is seen, consists of (a) preheater, (b) still of the ordinary type, (c) condensing worm, disposed in a water tank, (d) measuring tank, (e) storage tank, and (f) air compressor. The still is so arranged that it can either produce concentrated gas liquor, or the steam-ammonia stream can be sent to a saturator for the production of sulphate of ammonia. The still is capable of dealing with sufficient gas liquor to produce four tons of sulphate of ammonia per day. It can therefore be assumed from existing practice that the plant will produce six tons of concentrated gas liquor of 16 per cent NH_3 content per day. The preheater, which consists of alternate gas and liquor sections, made of cast-iron, has occasioned trouble. This is attributed to the action of hydrocyanic acid creating leaks in the tubes, with a consequent rapid weakening of the strong liquor. A multitubular heater-cooler of the type shown in fig. 126 is not to be recommended, as there is likely to be a diagonal travel of the gases, and certain of the tubes—particularly on the side where the cold gas liquor enters—are likely to remain at a low temperature, and thus aid corrosion. It is the author's experience that if a reasonable temperature can be maintained, the tubes of a heater-cooler are not likely to suffer from the action of hydrocyanic acid. A multiple flow cast-iron heater-cooler would certainly be preferable. It is said that the manipulation of the plant is peculiarly simple and affords reasonable flexibility. The operation can be discontinued or recommenced very readily; twenty minutes' preliminary steaming is said to be sufficient to prepare everything ready for use. A similar period of steaming enables the plant to be shut down.

The fixed ammonia content of the Paisley liquor constitutes a small proportion of the whole, and its extraction does not warrant the expense on lime and labour which would be necessary if the fixed ammonia had to be released.

SPECIAL METHOD OF WORKING

In certain districts of Great Britain it is evident that gas works will be compelled to consider the adoption of a special type of concentrated gas liquor plant, in order to obviate the creation of any spent liquor where the disposal of such is forbidden. The best method of procedure in such cases is to refrain from using milk of lime, and merely to dissociate the volatile ammonia, producing from it concentrated gas liquor. The spent liquor which still contains the fixed ammonia, after treatment in the way outlined later, is cooled and recirculated, preferably in the hydraulic main at the outset, and subsequently in the washers and scrubbers for the accumulation of further quantities of ammonia. This *modus operandi*, so far as the solution containing the fixed ammonia is concerned, can be pursued several times until the quantity of fixed ammonia is such as to warrant its despatch to the sulphate of ammonia or other such works where the concentrated gas liquor is treated.

It will be appreciated that from such works two qualities of liquor will arise: one which may be regarded as partially concentrated gas liquor, containing, say,

about 10 per cent of ammonia, and the other containing concentrated fixed ammonia of about 4 to 6 per cent ammonia content.

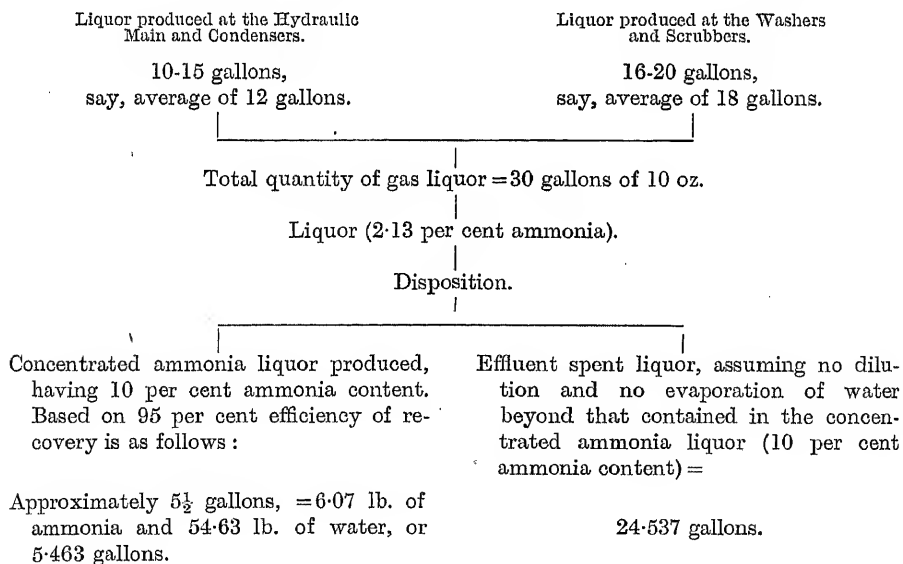
How far concentration can proceed depends on the amount of virgin liquor arising, on the extent to which water is used in the washers and scrubbers, and on the type of distillation plant adopted. Usually 10 to 15 gallons of virgin liquor are recovered per ton of coal carbonised.

At many works, 10 oz. (2·13 per cent NH_3) liquor is aimed at. At such works the average yield of gas liquor per ton of coal carbonised is 30 gallons.

If concentration of the gas liquor is to be effected in the manner outlined, it can only be undertaken in a boiler type of still, worked intermittently, in which the waste heat from the retort settings is used. The reason for the adoption of this type of plant is to regulate the water balance, so as to permit of working with a view to the avoidance of any effluent spent liquor.

WATER BALANCE, IN RELATION TO THE SPECIAL TYPE OF PLANT, AND METHOD OF WORKING

The following statement sets out the position as regards the water balance :



It is evident from the above that if the proposed method of working is to be applicable, it will be necessary to evaporate something of the order of 6·5 gallons of water for each ton of coal carbonised. This can be easily and inexpensively effected if the waste gases from the retort settings are used in the type of plant recommended.

After ammonia liquor of the required concentration has been produced, it will be necessary to continue the application of heat, but the condenser would be used

merely to condense the steam evaporated from the spent liquor containing the fixed ammonia. What is involved is this : the condenser would be provided with two outlets—one leading to the concentrated ammonia liquor storage boiler, and the other leading to the sewer. As soon as the volatile ammonia has been expelled from the liquor, then the outlet cock leading to the storage boiler would be shut, and the cock giving access to the sewer would be opened. Distillation would continue until the requisite quantity of water to admit of maintenance of the correct water balance had been removed.

It is certain that it would be profitable to adopt such an arrangement of plant and method of working at many of the gas works which are at present confronted with difficulties in the matter of the disposal of the effluent liquor, owing to stringent action on the part of County Councils and River Boards.

CHAPTER XII

CONCENTRATED AMMONIA LIQUOR AND LIQUOR AMMONIA

THE qualities of concentrated gas-liquor, concentrated ammonia liquor, and liquor ammonia constitute a subject which should be dealt with briefly at this juncture.

Concentrated gas liquor is, as its name implies, an impure ammoniacal liquor; indeed, one containing most of the hydrogen sulphide and carbonic acid and other volatile impurities of the original liquor, with about 16 per cent of ammonia. It is unwise to exceed the latter strength, owing to the large amount of carbonate present, which at higher strengths crystallises out (as has been explained in the previous chapter), dislocating the operation of the plant through blockages. In short, concentrated gas liquor is largely a solution of the sulphides and carbonates of ammonium.

Concentrated ammonia liquor consists for the most part of caustic ammonia. The usual specification to which the liquor has to conform is 25 per cent of ammonia, with not more than 0.5 per cent of hydrogen sulphide. When used in the manufacture of ammonium nitrate it is important that the total amount of cyanogen, calculated as ammonium thiocyanate, should not exceed 0.01 grm. per 100 c.c.

Colman and Yoeman, who had occasion during the war to examine a large number of samples of concentrated ammonia liquor from different plants in various parts of the country, have given some interesting figures having reference to the quality of this product (*vide Journal of the Society of Chemical Industry*, vol. 37, p. 319T, December 31, 1918). The information which they afford is so valuable that it is proposed to quote a certain portion of their contribution *in extenso*. They say: "Apart from the cyanogen compounds, the impurities found in appreciable quantity consist of the following:

"(1) *Hydrogen Sulphide*.—It is usually specified that the amount of this impurity shall not exceed 0.5 per cent, and in the majority of samples examined the quantity varies from this figure down to nil. A minority, however, contain much higher amounts, rising in one instance to 3.1 per cent, evidently owing to insufficient elimination of the hydrogen sulphide in the preheating process.

"(2) *Carbonic Acid*.—In many cases this is entirely absent, and is usually only present in small quantities, but in some instances up to 11 per cent of CO_2 was found. Such high amounts are always accompanied by excessive amounts of hydrogen sulphide, and are due to inadequate treatment of the crude liquor in the preheater. For most purposes the presence of carbonic acid in moderate amount is not in itself objectionable, but when it is present in considerable quantity the amount of other impurities is always high.

"(3) *Phenols*.—The samples always contain phenols, derived from those present in the crude liquor, which are partly volatilised with the steam in distillation. No systematic determination of the amount has been made, but in a few samples tested the quantity varied from 0.11 to 0.37 grm. per 100 c.c., averaging 0.23 grm.

"(4) *Pyridine*.—Pyridine bases are always present, the amount found in a few samples varying from 0.21 to 0.32 grm. per 100 c.c., averaging 0.26 grm.

"(5) *Ammonium Thiosulphate*.—The freshly prepared liquor is probably free from this impurity, which is formed secondarily by oxidation of the ammonium sulphides. The analytical methods tend to give results which are slightly too high, but the figures obtained from about fifty samples of different origin indicate that the amount varies from 0.08 to 0.35 gm. $(\text{NH}_4)_2\text{S}_2\text{O}_3$ per 100 c.c., no higher figure than the last having been obtained. These samples were, however, all tested within a few days of their manufacture, and the amount probably increases steadily when the liquor is stored with access of air, owing to the further oxidation of ammonium sulphides. At the same time, we are not aware that any complaint has been made of any prejudicial effect due to the presence of considerable quantities of ammonium thiosulphate in the liquor.

"CYANOGEN COMPOUNDS

"The especial point on which information was desired was the nature and amount of the cyanogen compounds present in samples of concentrated ammonia from different plants, these having been found of considerable disadvantage in the manufacture of ammonium nitrate, for which purpose it was desired that the total amount of cyanogen, calculated as ammonium thiocyanate, should not exceed 0.01 gm. per 100 c.c.

"In the liquor as first produced, the only cyanogen compound present appears to be ammonium cyanide, formed by the action of the hydrocyanic acid almost always given off with the ammonia from the crude liquor during distillation. In presence of ammonium sulphide, however, this ammonium cyanide rapidly undergoes further changes: as soon as the oxygen of the air gets access to the liquor, oxidation of the ammonium sulphides takes place, with formation especially of ammonium thiosulphate and polysulphides, the latter at once reacting with ammonium cyanide, forming the thiocyanate. Further, the ammonium cyanide solution produced acts on any iron (especially steel or wrought iron) with which it may come in contact, whether in the manufacturing plant or in the containing vessels, forming ammonium ferrocyanide. Hence, the liquor samples may contain ammonium cyanide, ferrocyanide, and thiocyanate, though the tendency is for the cyanide to disappear rapidly, owing to conversion into the other two forms, and in many cases ferrocyanide is absent, the only recognisable cyanogen compound being the thiocyanate.

"There is, however, a further source of thiocyanate other than the hydrocyanic acid evolved with the ammonia in the distillation, for it was found that, on standing, the total cyanogen content, in all forms of the liquor, undergoes a material increase. This is shown in the following series of tests made on the same sample of liquor after the lapse of different periods, the fresh sample being almost free from cyanogen derivatives:

"TABLE 44

				Total HCN, calculated as NH_4CNS .	
Freshly prepared product	.	.	.	0.0054	gram. per 100 c.c.
After standing 3 days	.	.	.	0.0061	„ „
„ 7 „	.	.	.	0.0167	„ „
„ 11 „	.	.	.	0.0175	„ „
„ 17 „	.	.	.	0.0228	„ „
„ 24 „	.	.	.	0.0304	„ „
„ 31 „	.	.	.	0.0304	„ „

"The cause of this increase was found to be the small quantity of carbon bisulphide usually present in the distillation gases from the still, and formed from the ammonium thiocarbonate always present in the crude liquor, this being absorbed in the concentrated liquor with the reformation of ammonium thiocarbonate. The latter, on standing, undergoes slow change, forming, among other products, ammonium thiocyanate. This change is shown by the following series of tests, in which a solution was made up of 100 parts of pure 25 per cent ammonia, 0.5 per cent of hydrogen sulphide, and 0.1 per cent of carbon bisulphide. If the whole of this carbon bisulphide were converted into ammonium thiocyanate, the amount of the latter in solution would amount to 0.1 per cent. Shortly after the preparation of the solution it was found that about one-fifth of the carbon bisulphide had already been converted into thiocyanate, and on standing the amount increased, until after about twenty days the thiocyanate remained constant, one-third of the carbon bisulphide having then been converted into thiocyanate :

"TABLE 45

				NH_4CNS .	
Fresh solution	.	.	.	0.0198	gram. per 100 c.c.
After standing 3 days	.	.	.	0.0228	„ „
„ 6 „	.	.	.	0.0258	„ „
„ 10 „	.	.	.	0.0304	„ „
„ 13 „	.	.	.	0.0314	„ „
„ 20 „	.	.	.	0.0334	„ „
„ 27 „	.	.	.	0.0334	„ „
„ 40 „	.	.	.	0.0334	gram. per 100 c.c."

As coke oven liquor contains larger amounts of ammonium cyanide than that of gas works, it is invariably found that the concentrated ammonia liquor produced at coke ovens contains a sensibly higher amount of cyanogen derivatives than that of gas works. Apart from objections to cyanides in the finished liquor, difficulty results by reason of the corrosive effect on iron, more especially wrought iron and steel. Many cases are known where the steel tubes of dephlegmators and condensers have developed serious leakages after a few weeks' operation, due to the action of cyanides.

Concentrated ammonia liquor of the above quality has for many years been manufactured in considerable quantity as an intermediate product in the manufacture of sodium carbonate by the ammonia-soda process.

Liquor ammonia is a much purer form of concentrated ammonia liquor. It should be colourless, even after exposure to light for some time. It should be clear and free from sulphides as revealed by lead acetate paper, and practically free from empyreumatic matter. The ammonia content varies between 25 and 30 per cent.

PLANT INVOLVING THE USE OF LIMED LIQUOR

In pre-war times the process in vogue at many chemical works for the production of concentrated ammonia liquor was one which involved the initial admixture of such a quantity of milk or cream of lime with the gas liquor to be distilled, as to arrest effectively the whole of the hydrogen sulphide and carbon dioxide which otherwise would be released in the distillation process.

Specially designed stills were employed, so as to obviate blockage in the trays constituting the still, due to the formation of calcium carbonate and calcium sulphide.

The ammonia discharged along with the steam from the still in this type of plant was practically free from H_2S and CO_2 . All that was necessary, therefore, was to employ a suitably designed dephlegmator of adequate size, so as to cool the gases to a temperature at which a dry, or approximately dry, ammonia gas was obtained, and to absorb the ammonia in purified town's water. The absorbers were generally arranged in the form of a cascade—three vessels being used. The water entered the higher vessel, and gravitated from vessel to vessel until the requisite strength of ammonia was obtained in the first, or lowest, vessel of the series.

These absorbers were arranged with water-cooling coils, so as to admit of efficient absorption of ammonia, by effectively dissipating the heat of formation of ammonium hydrate. The dephlegmator was usually so placed in relation to the still as to allow of the reflux liquor gravitating to the latter apparatus through a suitably sealed pipe.

It was particularly with this type of plant, an illustration of which is shown in fig. 127, that many ammonia distillers were familiar thirty years ago.

The "colonne inobstruable," to which reference has been made in Chapter VI., was found to be especially adapted to the use of limed liquor in the production of concentrated ammonia liquor and liquor ammonia. Subsequently, the principle of desulphurisation and decarbonation of gas liquor, which had been known for some time, and which had been used at a few gas works in the United Kingdom in the process of liquid purification, was resorted to.

Observations concerning the development of the latter process will be made in Chapter XVI.

It is, however, important that consideration should be given at once to the underlying principles involved in the desulphurisation and decarbonation of gas liquor by heat, to the design of dissociators, and to the advantages of the heat application method as contrasted with the removal of hydrogen sulphide and carbonic anhydride by liming.

A. Lime and Liquor Mixer.
B. Free Still.
C. Fixed Still.
D. Dephlegmator.
E_{1,2,3}. Absorbers.
F. Storage Boiler.
G. Water Scrubler.

a. Twin Strainer.
b. Centrifugal Pump
c. Cooling Coils
a₁. Water Inlet.
a₂. Water Outlet.
e. Reflux.
f. Gas Liquor Feed.
g. Milk of Lime Feed.

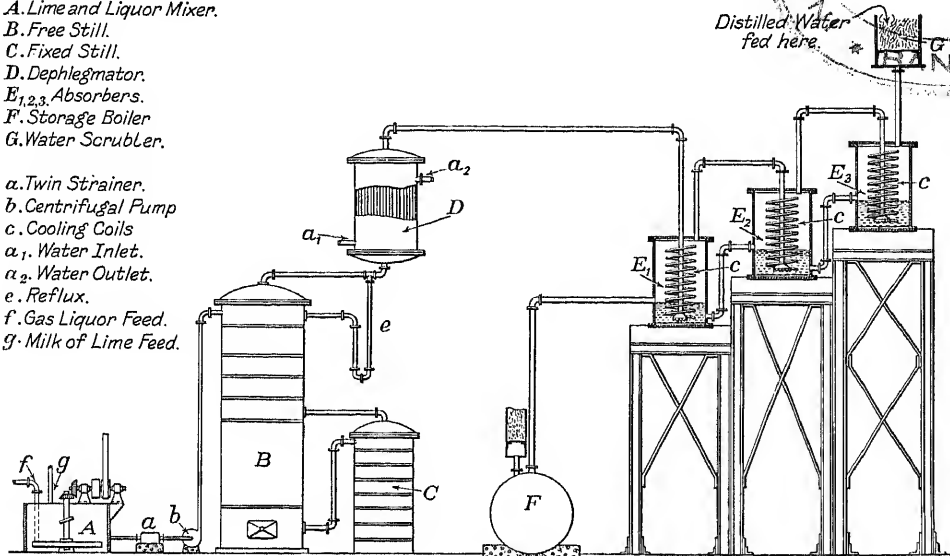


FIG. 127.—DIAGRAMMATIC SKETCH OF LIQUOR AMMONIA PLANT.

THE UNDERLYING PRINCIPLES OF THE DESULPHURISATION AND DECARBONATION OF GAS LIQUOR BY HEAT

The original process of C. Hills (B.P. 5874, of 1884) takes advantage of the properties of carbonate and sulphide of ammonium. The ammonia, hydrogen sulphide, and carbon dioxide present in gas liquor are in chemical combination, and as such, the individual tensions of the latter compounds are invariably less than when they exist in the free state. The application of heat to about 80° C. results in partial disunion, and the three gases cited then largely exist in solution—not as carbonate, sulphide, and hydrosulphide, but (in conformity to Henry's law) as free gases soluble in water under equal conditions.

The solubility of ammonia in water in like condition is appreciably greater than H₂S and CO₂. Hence, the latter acid gases are dispersed, while the ammonia is held in solution. As, however, the less soluble gases are evolved at a temperature at which water has a comparatively high vapour pressure, a considerable volume of water vapour is removed, together with a small volume of ammonia in solution.

The author's belief is that the inventors of the process for the desulphurisation and decarbonation of ammoniacal liquor by the application of heat *via* steam, made a rather extravagant claim for their process. The temperature at which Hills worked was about 80° C. Lunge, in his book on *Coal Tar and Ammonia*, states that if gas liquor is heated by steam to a temperature a little below boiling-point the whole of the H₂S and CO₂ is expelled with little loss of ammonia.

The author's experience suggests that these statements are not strictly correct,

and need qualification. The faulty design of many concentrated ammonia liquor plants installed during the War was in part attributable to an imperfect conception of what was involved.

DESIGN OF THE DISSOCIATOR

Extended practical experience reveals that extreme care must be taken in the design of the dissociator and the other apparatus working in conjunction with it. The author has demonstrated that only a small range of temperature exists between the dissociation point of carbonate and sulphide of ammonium, and the boiling-point of water (*vide* Parrish, *Journal of the Society of Chemical Industry*, vol. 37, p. 232T). It is seen from the table below that water under a pressure of 2.5 in. mercury above atmospheric pressure boils at 260.3° F., or under a pressure of 4.7 in. mercury gauge above atmosphere boils at 219.6° F.

TABLE 46

Total Pressure per sq. in., measured from a Vacuum (Absolute Pressure).	Pressure above Atmosphere.	Sensible Temperature in Degrees Fahrenheit.	Pressure in ins. Mercury Gauge above Atmosphere.
lb.	lb.		
14.7	0.0	212.0	0.0
15.0	0.3	213.1	0.6
15.5	0.8	215.7	1.5
16.0	1.3	216.3	2.5
16.5	1.8	217.9	3.6
17.0	2.3	219.6	4.7
17.5	2.8	221.0	5.8
18.0	3.3	224.4	6.9

Similarly, the author's practical experience of the operation of large concentrated ammonia liquor plants leads to the view that if the boiling-point of gas liquor represented by the absolute pressure is attained, desulphurisation and decarbonation will be practically complete, and the resulting caustic ammonia produced will, when concentrated by dephlegmation, give a product of a quality which will fulfil the usual standard specification.

The attainment of the boiling-point under the conditions indicated is more difficult than would appear at first sight, and necessitates a special type of dissociator.

It should be remarked that dissociators can be worked at a few lb. pressure per square inch, or at 20 to 50 lb. pressure per square inch. Herein lies a fundamental difference which will be enlarged upon shortly.

DIRECT AND INDIRECT STEAM

If the dissociator is designed to work at comparatively low pressures, then the question of the relative advantages and disadvantages of direct and indirect steam as a heating media need to be considered. A quantity of direct steam as a carrier is undoubtedly essential, but if more effective dissociation of the ammonium compounds of the gas liquor is desired, the use of a relatively higher percentage of indirect steam is a desideratum.

Direct steam increases the volume of the vapour with which the dissociator has to deal, and generally speaking, with the usual types of plant with which the country was familiar during the War, the temperature of the liquor can only be increased at the expense of pressure. Thus one reaches a point at which one condition (increase of pressure) more than nullifies the other (increase of temperature). On the other hand, indirect steam is more likely to promote an increase of temperature without proportionately increasing the pressure, and hence, more effective dissociation is attained.

This leads to the view that in the design of the trays of a dissociator the surface area for the ascent of the gases, and the space for the descent of the liquor in the succeeding trays, starting from the top, should progressively increase in proportion to the computed volume of gases, etc., existing at each tray.

As experience of this section of the plant clearly discloses, the liquor on the lower tray is often out of pressure equilibrium with the liquor on the tray above, with the result that the liquor on the upper tray is prevented from flowing through the overflow arrangement provided. Accordingly, the liquor accumulates in the second tray, immersing the hoods, frogs, or bubblers (whatever device for the distribution of the gases is arranged), and there is a sort of battle royal between the pressure of the gases and the accumulation of liquor. Occasionally the liquor will accumulate to such a height in the dissociator as to overcome the gaseous pressure, and the dislocation will adjust itself. More often than not, however, the direct steam has to be reduced, or shut off, to allow of a reduction of pressure, and the establishment of pressure equilibrium when the liquor will again commence to flow.

At these times of derangement, when there is an unequable or erratic flow of liquor, inefficient dissociation of the ammonium compounds arises.

DISSOCIATOR EQUIPMENT—STEAM COILS

Steel coils are not to be recommended, as they are attacked by the cyanide compounds of the gas liquor, as has already been remarked at an earlier part of this chapter. Lead coils are not so susceptible to chemical action, but they are likely to burst. Cast-iron coils are undoubtedly to be recommended, as they can be made to withstand the steam pressure, and are immune from chemical action.

Julius Pintsch, Aktien-Gesellschaft, of Berlin, provides for a dissociator in German Patent No. 178030, which contemplates the use of steel coils, but judging from the drawing, the gas spaces are not proportioned to meet the varying volumes of gases to be dealt with. Fig. 128 represents a sectional elevation of the dissociator,

the lower part of which is a heating chamber, of which fig. 129 furnishes a horizontal section. The upper portion of the still is merely a series of tray sections with gas distribution devices and liquor seals arranged for arresting any ammonia carried forward with the gases by means of cool gas liquor. In the bottom chamber, where

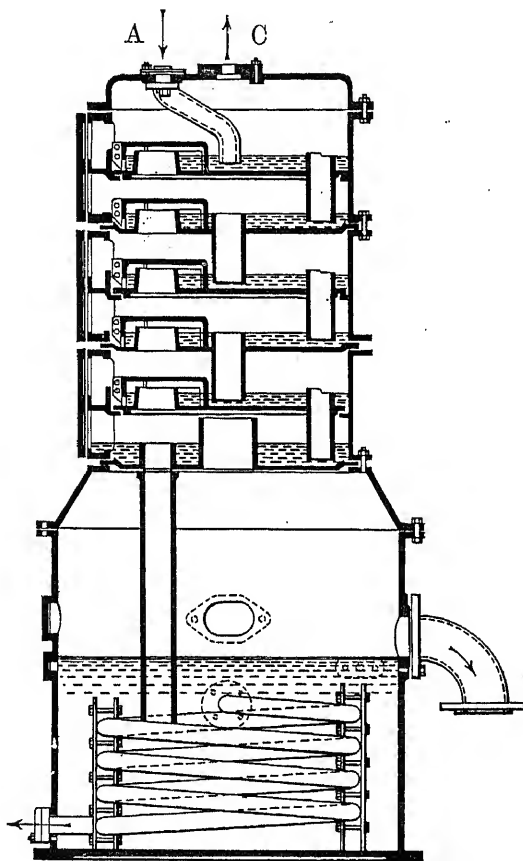


FIG. 128.—PINTSCH STILL.

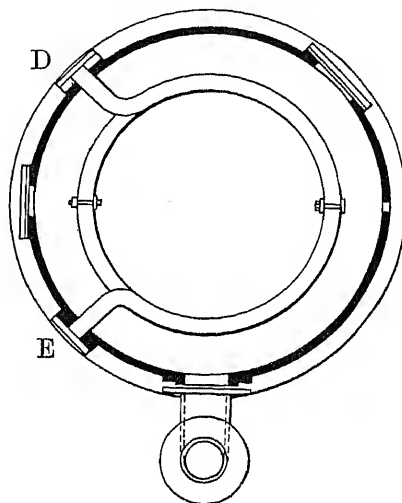


FIG. 129.—PINTSCH STILL.

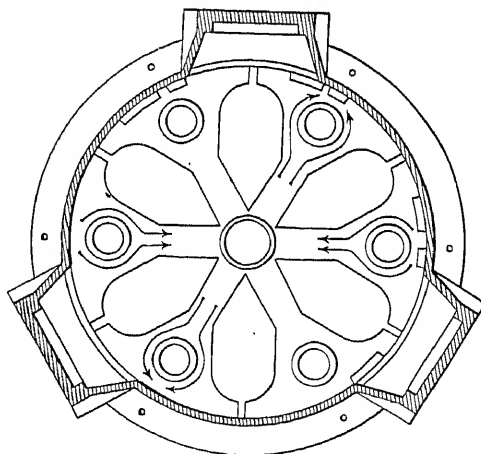


FIG. 130.—PINTSCH STILL.

the heat is applied, it will be seen that the steam enters at D, and leaves the worm in the form of condensed water at E.

Fig. 130 shows a horizontal section through the absorption segment of the still. The gas liquor to be desulphurised and decarbonated is fed to the still at A, and the waste gases leave at C.

A COMPLETE PLANT FOR THE MANUFACTURE OF CONCENTRATED AMMONIA LIQUOR WITH LOW PRESSURE DISSOCIATOR

Fig. 131 shows a complete arrangement of plant for the manufacture of concentrated ammonia liquor, which was in operation during the War. The dissociator is of the Brunner, Mond type, worked at a comparatively low pressure, and, as will be seen from the illustration, consists of four heating trays, each containing 15 inches of liquor, and one cooling tray, in connection with which water can be used through the coil provided.

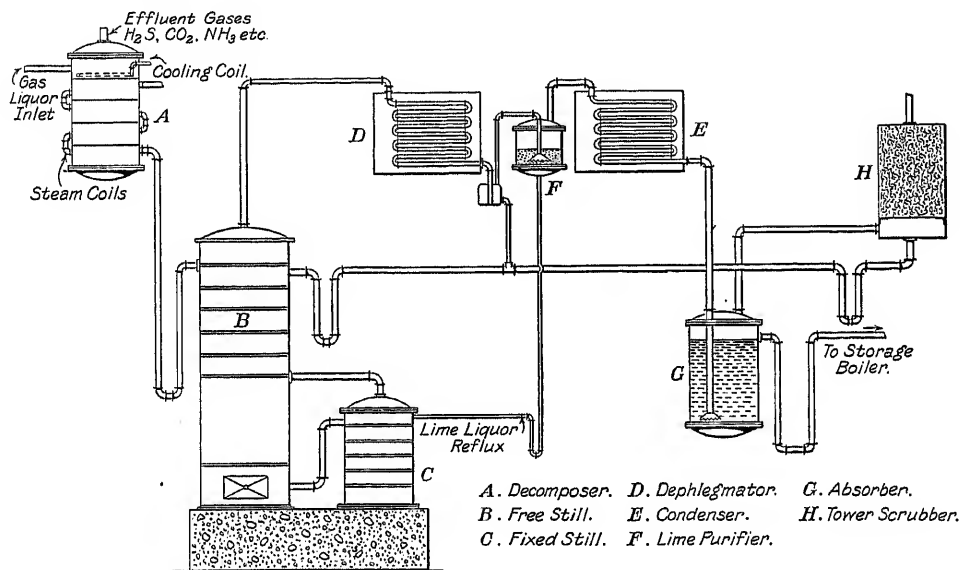


FIG. 131.—DIAGRAMMATIC SKETCH OF LIQUOR AMMONIA PLANT.

The application of heat is by indirect steam through the separate coils of the four heating trays, and direct steam to the bottom tray. A plant similar to the one shown was in operation at one of the coke ovens in the Midlands, and was the subject of comment in a Presidential Address by John W. Lee (*vide Gas World*, Coking Section, December 1, 1917).

The plant was designed to deal with thirty to thirty-five thousand gallons of gas liquor per day, containing from 1.0 to 1.2 per cent of ammonia.

The liquor is fed to the top tray of the dissociator by gravity from an overhead tank, and is maintained at a constant level. The temperatures to which the liquor is subjected on the several heating trays and the respective contents of ammonia (free and fixed), carbon dioxide and hydrogen sulphide, are also furnished, and merit consideration. The tests in question are subjoined.

LIQUOR TO DECOMPOSER.

TABLE 47

Test No. 1

Steam pressure on main . . . 140 lb.
 Steam pressure on decomposer . . . $9\frac{1}{4}$ lb.
 Direct steam on tray No. 5.

	Temp. °C.	Ammonia.			CO ₂ .	H ₂ S.
		Free.	Fixed.	Total.		
Feed	40	0.976	0.238	1.214	0.973	0.126
No. 1 tray . . .	39	0.976	0.236	1.212	0.946	0.120
„ 2 „	47	1.059	0.204	1.263	0.940	0.116
„ 3 „	80	1.051	0.197	1.248	0.529	0.102
„ 4 „	$94\frac{1}{2}$	0.964	0.190	1.154	0.526	0.095
„ 5 „	100	0.953	0.186	1.139	0.132	0.068

Gas from decomposer to foul main free from NH₃. “Lime liquor” from lime vessel to stills contained 14.50 per cent NH₃. “Run back” from the first condenser contained 3.40 per cent NH₃, 0.48 per cent H₂S, and 0.46 per cent CO₂. The waste liquors from the stills contained: No. 1 still, 0.0170 per cent NH₃; No. 2 still, 0.0019 per cent NH₃; No. 3 still, 0.0281 per cent NH₃. Concentrated liquor contained 25.16 per cent NH₃ and 0.58 per cent H₂S.

TABLE 48

Test No. 2

Steam pressure on main . . . 130 lb.
 Steam pressure on decomposer . . . $8\frac{1}{2}$ lb.
 Direct steam on tray No. 5.

	Temp. °C.	Ammonia.			CO ₂ .	H ₂ S.
		Free.	Fixed.	Total.		
Feed	0.976	0.146	1.122	1.320	0.143
No. 1 tray . . .	43	0.978	0.146	1.124	1.300	0.139
„ 2 „	46	1.013	0.133	1.146	0.647	0.129
„ 3 „	68	1.286	0.127	1.413	0.500	0.119
„ 4 „	93	1.309	0.196	1.505	0.110	0.119
„ 5 „	$99\frac{1}{2}$	0.836	0.167	1.003	0.107	0.045

CONCENTRATED AMMONIA LIQUOR AND LIQUOR AMMONIA 205

Gas from decomposer to foul main free from NH_3 . The waste liquors from stills contained : No. 1 still, 0.0022 per cent NH_3 ; No. 2 still, 0.0087 per cent NH_3 ; No. 3 still, 0.0298 per cent. Concentrated liquor contained 24.47 per cent NH_3 , and 0.44 per cent H_2S .

TABLE 49

Test No. 3

Steam pressure on main . . . 140 lb.
Steam pressure on decomposer . . . $10\frac{1}{2}$ lb.
No direct steam on No. 5 tray.

	Temp. °C.	Ammonia.			CO_2 .	H_2S .
		Free.	Fixed.	Total.		
Feed	$40\frac{1}{2}$	1.058	0.258	1.316	0.996	0.158
No. 1 tray . .	39	1.058	0.258	1.316	0.946	0.152
" 2 " . .	43	1.027	0.248	1.275	0.646	0.102
" 3 " . .	64	1.115	0.250	1.365	0.529	0.100
" 4 " . .	$90\frac{1}{2}$	1.197	0.268	1.365	0.506	0.096
" 5 " . .	98	1.296	0.286	1.582	0.168	0.090

Gas from decomposer to foul main free from NH_3 . The waste liquors from the stills contained : No. 1 still, 0.010 per cent NH_3 ; No. 2 still, 0.0029 per cent NH_3 ; No. 3 still, 0.0204 per cent NH_3 . Concentrated liquor contained 25.34 per cent NH_3 , and 0.40 per cent H_2S .

The figures are self-explanatory, and need no comment, except to point out that it is impossible to produce a concentrated liquor with a sulphuretted hydrogen content of 0.5 per cent, on this type of plant, without liming.

It is interesting now to examine these analyses. Take Test No. 1. Assuming no attempt had been made to remove the H_2S from the gas liquor, the quantity of this impurity in the final liquor, containing 25.16 per cent NH_3 , would be :

$$\frac{25.16}{1.214} \times 0.126 = 2.61 \text{ per cent } \text{H}_2\text{S}.$$

Examine now the distribution of this impurity as a result of the working of the plant :

TABLE 50

	Per cent H_2S .	Per cent Efficiency.
By calculation one finds that 1.108 is removed by the dissociator .	42.1	
" " " 0.922 is removed by the lime purifier .	35.3	
" " " 0.580 remains in the final liquor .	22.2	
	<u>2.610</u>	<u>99.6.</u>

Take Test No 3, which reveals slightly better working. Again assuming no attempt had been made to purify the resultant concentrated ammonia liquor from H_2S , the final 25.34 per cent of ammonia would contain 3.04 per cent of H_2S .

TABLE 51

	Per cent H_2S .	Per cent Efficiency.
By calculation one finds that 1.60 is removed by the dissociator		52.6
" " " 1.04 is removed by the lime purifier		34.2
" " " 0.40 remains in the final liquor		13.1
	<u>3.04</u>	<u>99.9</u>

It is seen from the above calculations that the efficiency of the dissociator is respectively 42.10 per cent and 52.60 per cent, or, in other words, assuming there was no lime purifier, the concentrated ammonia liquor in the respective cases (those where the ammonia content is 25.16 per cent and 25.34 per cent) would contain 1.502 per cent and 1.44 per cent of H_2S .

It should be remarked at this juncture that the caustic liquor from the dissociator enters the ammonia still, and the steam and ammonia stream emerging therefrom enters the dephlegmator. The reflux from the dephlegmator returns to the still, entering the second section from the top. The ammonia gas, etc., enter the liming vessel, bubbling through a seal of milk of lime, as shown. After liming, the ammonia gas, etc., pass to the condenser, thence to the absorber, and to the storage tank. The milk of lime, after use in the liming vessel, brings with it a certain quantity of ammonia, and enters the top section of the fixed still.

There are two or three undesirable features in this arrangement of plant to which attention should be directed.

It will be realised that the dissociator is incapable of giving a caustic liquor sufficiently free from H_2S to produce a final liquor complying with the specification so far as hydrogen sulphide content is concerned. Hence it is necessary to resort to washing the gas with milk of lime so as to fix a further portion of the H_2S , and incidentally, an additional quantity of CO_2 . A sensible quantity of ammonia will inevitably be absorbed by the limed liquor, and as the latter is led to the fixed still for treatment, extra work is imposed on this section of the ammonia distillation plant. It will therefore be appreciated that the effluent liquor will suffer, because it will be impossible to reduce the ammonia content to the usual limit otherwise obtaining. Moreover, additional steam is required to release again the ammonia fed to the fixed section *via* the limed liquor.

All these points emphasise the need for a well-designed and efficient dissociator, if the use of undue quantities of steam is to be avoided, and loss of ammonia *via* the effluent liquor is to be averted.

It is the author's belief that improvement can only come by the adoption of dissociators working under greater pressure than 2 to 5 lb. above atmospheric.

HIGH PRESSURE DISSOCIATORS

The underlying advantage of high pressure dissociators is one which was disclosed by the late William Young, of Peebles, who took out a patent in 1885 for "Improvements in the Preparation of Caustic Ammonia." A debt of gratitude is due to Young for having indicated the direction in which development should proceed. Young was cognisant of the fact that at atmospheric pressure the range of temperature between the boiling-point of water and the dissociation-point of carbonate and sulphide of ammonium was very small. In addition, he appreciated that if dissociation is conducted under considerable pressure, 20 to 50 lb. in excess of atmospheric, a greater quantity of caustic ammonia can be held in solution by the water, while the dissociation of carbonate and sulphide of ammonium is not materially affected.

Herein lies the basic principle of a method which is calculated to give tolerably pure caustic ammonia.

The process of desulphurisation and decarbonation by heat at high pressure has been in operation at one or two works in this country, and has been found to give eminently satisfactory results. Fig. 132 shows a diagrammatic representation of the plant named, which embraces (a) a dissociator, (b) receivers in the form of boilers for the absorption of the ammonia associated with the H_2S and CO_2 , etc., released as gas liquor, (c) a finishing still to which the caustic liquor is fed by gravity and the

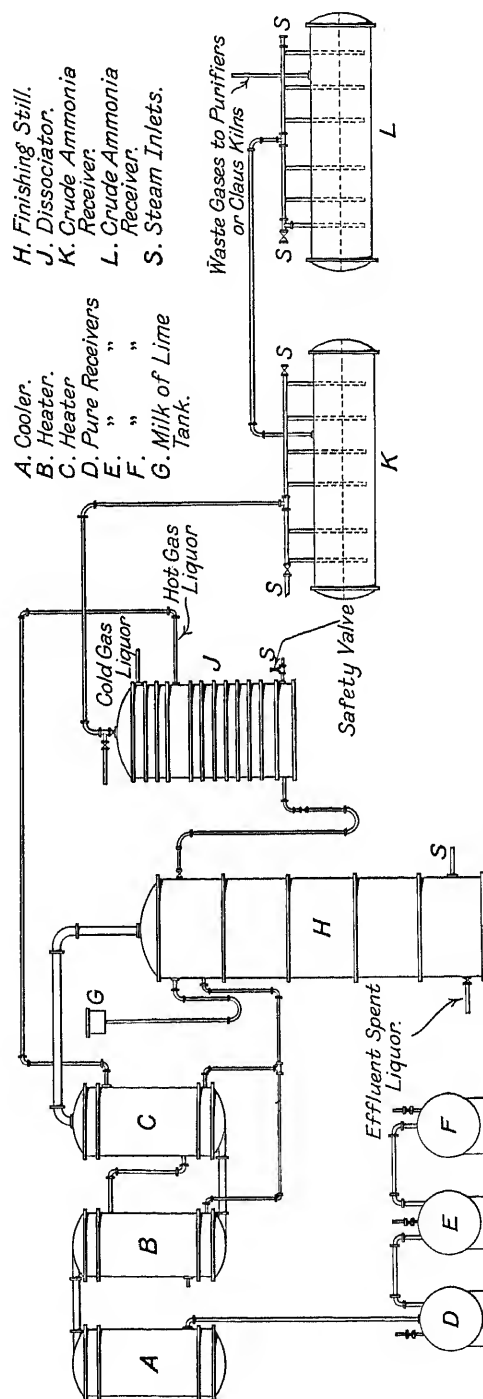


FIG. 132.—LIQUOR AMMONIA PLANT WITH HIGH PRESSURE DISSOCIATOR.

gases emerging from the top of which are passed to (d) two tubular single flow dephlegmators and (e) a condenser, and (f) special receivers are used for the absorption of the pure, dry ammonia in the case of liquor ammonia manufacture, or when concentrated ammonia liquor is desired, the temperature at the outlet of the dephlegmator is controlled in such a way as to admit of sufficient water vapour passing along with the ammonia to give the required strength of the product.

The outlet from the dissociator consists of a 2 in. main provided with a cock.

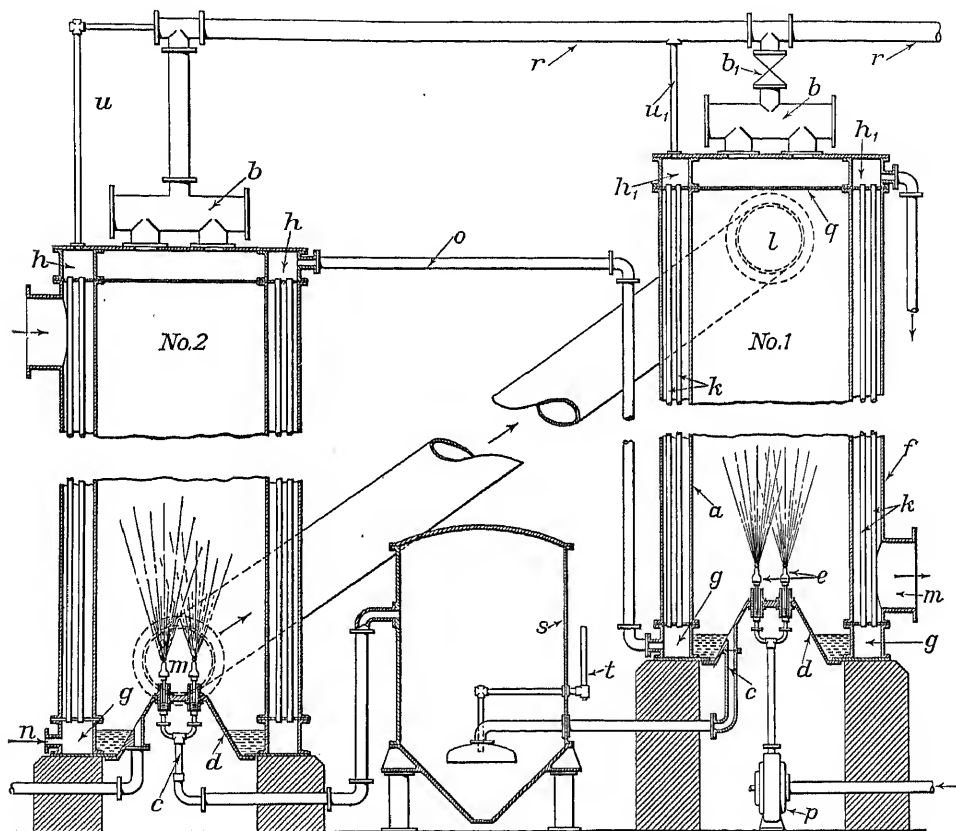


FIG. 133.—WYLD'S JETS.

By its operation, it will be realised that the predetermined pressure at which it is proposed to work the dissociator can be attained.

It is important with any such plant, where there is a tendency for blockage to occur due to the crystallisation of ammonium bicarbonate, that an efficient type of safety valve of adequate size should be provided on the steam pipe to the dissociator. It is futile to place the safety valve on the top of the dissociator, as owing to the possible crystallisation of ammonium bicarbonate it will be impossible for the safety valve to function properly.

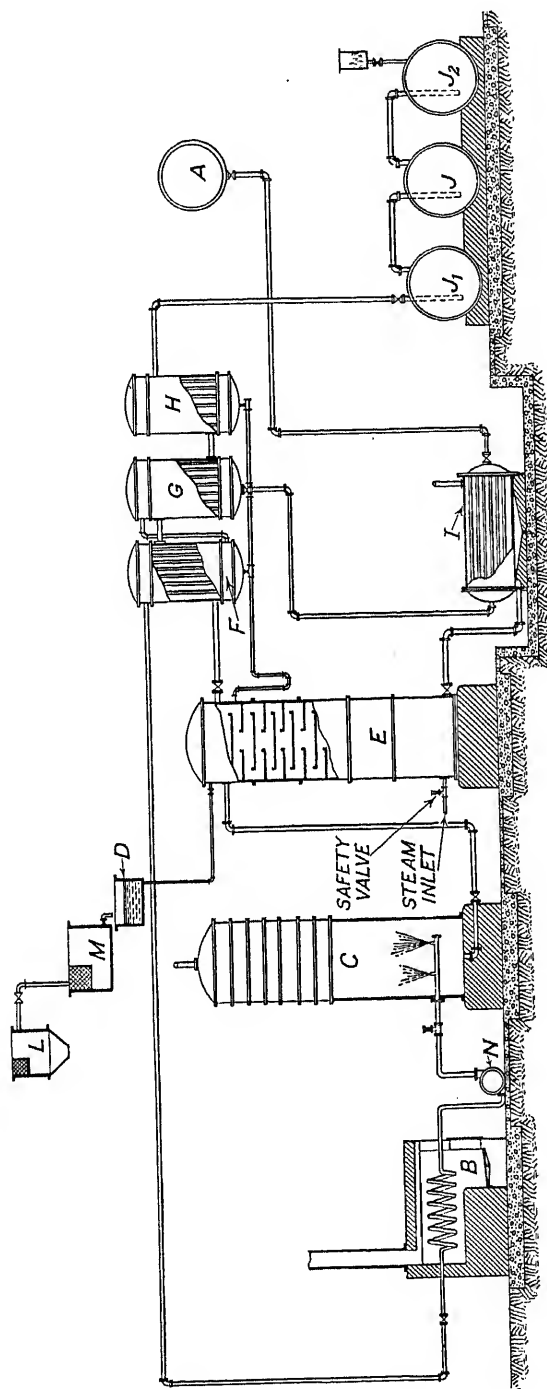


FIG. 134.—LIQUOR AMMONIA PLANT SHOWING COIL PREHEATER.

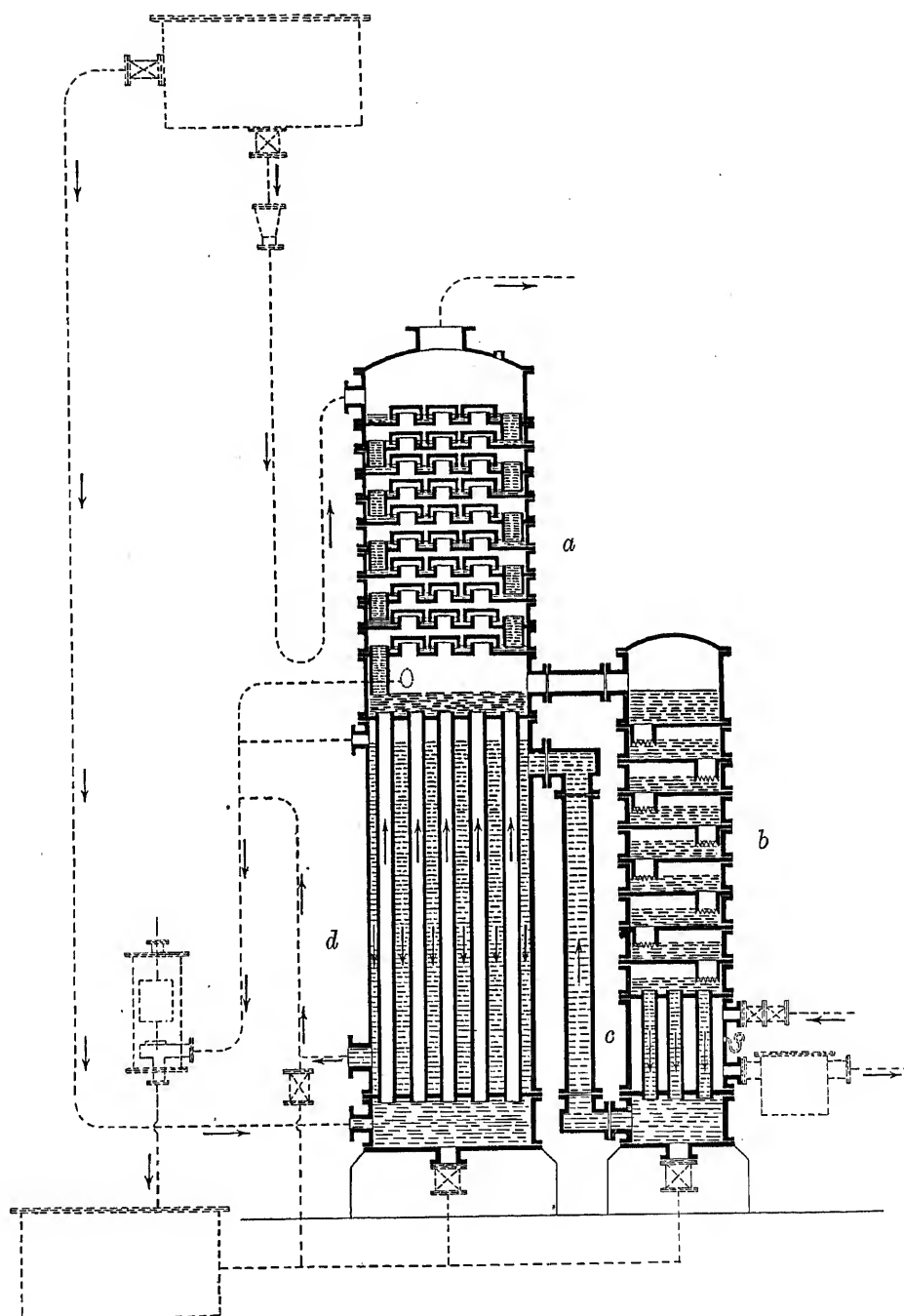


FIG. 135.—FABRY'S STILL.

The dissociator is placed at a higher level than the finishing still. In this way, due to the pressure under which the dissociator is worked, it is possible to discharge the caustic ammonia leaving the dissociator to the finishing still without having recourse to pumping. This is a distinct advantage, as loss of ammonia, which invariably arises at the pump glands when hot caustic liquor has to be pumped, is obviated.

A further improvement in the form of a high-pressure dissociator was introduced by W. Wyld about fourteen years ago (B.P. 25716, 1910). Fig. 133 shows this type of dissociator, which provides for spraying the liquor into a void chamber, which is worked at a pressure of 25 lb. per square inch above atmospheric, by the operation of a cock or valve in the waste gas outlet pipe. The gas liquor is usually preheated by interchange heat, and is then pumped through a series of 2-in. coils, which are heated by the products of combustion from a coke fire to a temperature of 260° F. Hot liquor is sprayed through two or three Korting's sprays having 14-mm. orifices. Due to the fine atomisation and by reason of the relative solubilities at the high temperature prevailing, the hydrogen sulphide and carbon dioxide are released, and a tolerably pure caustic ammonia is condensed and can be run by the inherent pressure of the dissociator to the finishing still.

A complete plant for the manufacture of concentrated ammonia liquor or liquor ammonia, embracing a coil preheater and spray dissociator, is shown in fig. 134. This arrangement of plant is intended to reduce the steam consumption to a minimum, not only by taking advantage of the potential heat of the effluent spent liquor and part of the heat of the steam-ammonia stream leaving the finishing still, but also by the maintenance of a temperature in the dephlegmator slightly below the dew-point, so as to reduce the amount of ammonia returned to the finishing still *via* the reflux liquor, to a minimum.

This is an all-important point if the steam consumption is to be kept at a low figure.

OTHER TYPES OF DISSOCIATORS

Réne Fabry, in his patent (No. 317, 1914), appears to have directed his attention to the economy of steam in the production of ammonia liquor. In general, the dissociator consists of four distinct main parts, either joined direct or connected to each other by suitable piping. Fig. 135 represents this type of apparatus.

The four principal parts are (a) the dephlegmator or scrubber, (b) the dissociator, (c) the superheater, and (d) the preheater. The operation of the plant is as follows: The bulk of the crude liquor is fed into the bottom of the preheater. It ascends the tubes in counter-current flow to the caustic liquor, which descends on the outside of the tubes. On reaching the void tower chamber at the top of the preheater, the liquor (which, by this time, is practically at boiling-point) meets that portion of the crude liquor which has been fed to the top of the dephlegmator, or absorber. The supply of cold liquor to the dephlegmator is regulated, so as to maintain a sufficiently low temperature to ensure the re-absorption of the ammonia which is associated with the H_2S and CO_2 . The whole volume of hot liquor flows

into the dissociator at the top, and passes downwards through the several bubbling

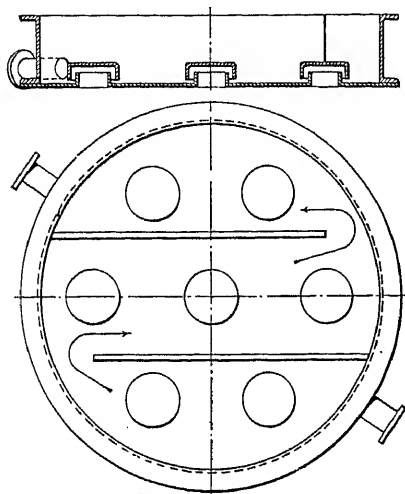


FIG. 136.
ELEMENTS OF DESULPHURISING STILL.

devices, in which it is subjected to the heat and movement of the steam and gases generated in the superheater. The steam admitted to the superheater passes around the tubes through which the liquor travels on its way to the preheater. Cooling of the dissociated liquor is effected by the heat interchange which takes place in the preheater.

A feature of this apparatus is that indirect steam is used, and that the separation of the several functions of the plant admits of better control.

Fig. 136 shows the elements of a dissociator which has been found to operate very satisfactorily. The underlying idea is to secure maximum travel of the liquor, and to provide a fairly large area for the ascent of the gases. Such provision is eminently desirable, and is calculated to avoid an

erratic flow of liquor due to an absence of equilibrium, to which attention has already been directed.

INITIAL LIMING OF THE GAS LIQUOR V. DESULPHURISATION AND DECARBONATION BY THE APPLICATION OF HEAT

Where the contents of hydrogen sulphide and carbon dioxide are large—say, of the order of 0.4 per cent and 2 per cent respectively, based on gas liquor containing 1.73 per cent NH_3 —it will be realised that an appreciable quantity of additional lime has to be used, if the impurities are to be fixed in this way. Moreover, if blockages of the still, by reason of calcium carbonate, are to be obviated, it is necessary to adopt a rotary still of the Mallet type, or otherwise to design the elements in such a way as to avoid blockage. It will also be appreciated that where the practice of liming is followed hydrogen sulphide is lost, whereas in desulphurisation and decarbonation of gas liquor by the application of steam, sulphuretted hydrogen is available for use in the manufacture of sulphuric acid, or can be recovered as Claus sulphur, or as a cruder form of sulphur in spent oxide.

The introduction of the additional lime, even as cream of lime, for the fixation of the impurities named, represents a fairly appreciable dilution of the liquor, and obviously involves the use of an increased quantity of steam. Moreover, with a continuously driven rotary still, there is the consumption of extra steam or power for driving.

In the production of caustic ammonia from gas liquor by the application of direct steam in a low pressure dissociator, it is certain that a sensible amount of

dilution takes place, ranging from 15 to 25 per cent. Additional apparatus is also involved as contrasted with that required when the H_2S and CO_2 are fixed by means of milk of lime.

On the whole, the author would recommend the adoption of a high-pressure type of dissociator, rather than the fixation of the hydrogen sulphide and carbonic anhydride by milk or cream of lime.

WORKING AND CONTROL OF CONCENTRATED AMMONIA LIQUOR, AND LIQUOR AMMONIA STILL'S AND PLANT

Nothing aids intelligent working and judicious control of any chemical plant more than a correct appreciation of the underlying principles involved. If one can clearly visualise what is occurring in the various parts of the plant, and what is required, then given the necessary diligence, good results can be expected.

Before proceeding to indicate the several points to be observed in the working and control of plants for the manufacture of the two products in question, it is proposed to furnish some data which, it is hoped, will be helpful in enabling one to envisage these operations in a way which perhaps has not been possible before.

The following diagram, fig. 137, shows the ammonia concentration in the gas

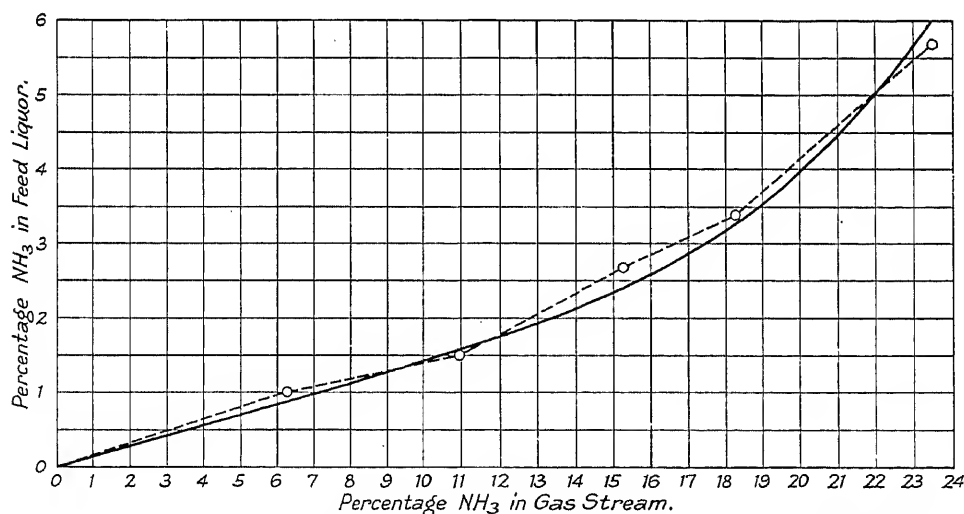


FIG. 137.—GRAPH SHOWING NH_3 CONCENTRATION IN GAS STREAM WITH VARYING CONTENTS OF NH_3 IN FEED LIQUOR.

stream with varying contents of ammonia in the feed liquor. This graph represents determinations made on an experimental plant, whereas those given in Chapter IV. are arrived at as the result of large scale operations.

Basing on the ammonia concentration in the gas stream peculiar to a given

TABLE 52

NH ₃ in Feed Liquor.	1 per cent.			1.5 per cent.			2 per cent.			2.5 per cent.			3.0 per cent.			3.5 per cent.			4.0 per cent.		
	Steam.		Per cent.	B.Th.U.		Per cent.	B.Th.U.		Per cent.	B.Th.U.		Per cent.	B.Th.U.		Per cent.	B.Th.U.		Per cent.	B.Th.U.		Per cent.
	lb.	2-12	11.8	2044	2-12	16.5	2044	2-12	20.8	2044	2-12	23.9	2044	2-12	26.4	2044	2-12	28.9	2044	2-12	30.6
Heat required for dissociation of 1 lb. of NH ₃	2970	3-08	17.1	1965	2-04	15.8	1470	1-53	15-0	1170	1-21	13-6	972	1-01	12-6	828	0-86	11.7	720	0-75	10.8
Heat required to raise X-lb. H ₂ O from 88.2° C. to 105° C.	380	0-39	2.1	380	0-29	3.2	380	0-39	3-8	380	0-39	4.4	380	0-39	4.9	380	0-39	5.3	380	0-39	5.6
Heat required to raise 6 lb. H ₂ O (with Radiation loss) from 70° C. to 105° C.	800	0-83	4.5	800	0-83	6.1	800	0-83	8.1	800	0-83	9.4	800	0-83	10.4	800	0-83	11.3	800	0-83	11.9
Heat equivalent of Y-lb. H ₂ O in condensate (containing N per cent NH ₃ and M per cent H ₂ S, etc.)	11-60	64.5	..	7-50	58.1	..	5-34	52.3	..	4-32	48.7	..	3-66	45.7	..	3-14	42.8	..	2-85	41.1
Total	18-02	100.0	..	12.88	100.0	..	10.21	100.0	..	8.87	100.0	..	8.01	100.0	..	7.34	100.0	..	6.04	100.0

VALUES OF X, Y, N, AND M

X	99.0	65.5	49.0	39.0	32.4	27.6	24.0
Y	11.6	7.5	5.34	4.32	3.66	3.14	2.85
N	7.3	10.4	13.4	15.5	17.3	19.0	20.1
M	8.2	11.7	15.2	17.5	19.5	21.4	22.7

strength of gas liquor, computations have been made showing the British Thermal Units, lb. of steam, and relative percentages involved for a particular strength of gas liquor.

With the aid of the above data and knowledge of the conditions obtaining in the production of concentrated ammonia liquor, fig. 138 has been prepared. Proceeding in a similar way, but in this case representing the conditions known to exist in the manufacture of liquor ammonia, where a dry ammonia gas is produced, fig. 139 has been prepared.

Fig. 138 enables one to follow the disposition of the ammonia and water

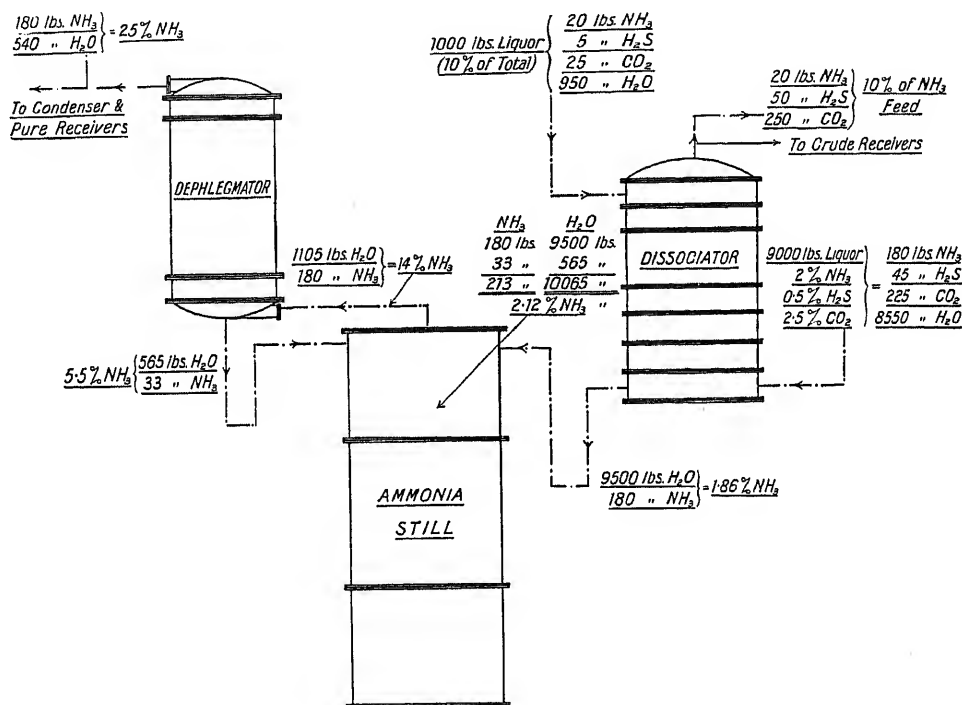


FIG. 138.—DISPOSITION OF AMMONIA AND WATER IN CONCENTRATED AMMONIA LIQUOR MANUFACTURE.

entering the dissociator as hot gas liquor, and leaving this apparatus for the finishing still as caustic ammonia; also, the quantity of ammonia, hydrogen sulphide, and carbon dioxide passed from the dissociator as waste gases after these have been washed with 10 per cent of cold gas liquor, which introduces a further quantity of ammonia and water, etc.

The caustic ammonia passes to the finishing still, and after a time equilibrium is attained, and a closed cycle of conditions is established. It is seen that the concentration of ammonia in the feed liquor to the finishing still is raised from 1.86 per cent to 2.11 per cent. This is due to the introduction of the reflux liquor from the dephlegmator to the still. An ammonia content in the gas liquor of 2.11

per cent is equivalent to an ammonia concentration in the steam-ammonia stream of 14 per cent.

From this it will be seen that with the quantity of gas liquor undergoing treatment, 1105 lb. of water and 180 lb. of ammonia are passing to the dephlegmator. As 25 per cent concentrated ammonia liquor is equivalent to a ratio of 540 lb. of water : 180 lb. of ammonia, it is clear that 565 lb. of water must be returned to the finishing still as reflux liquor.

This water, of course, cannot return without bringing along with it a certain

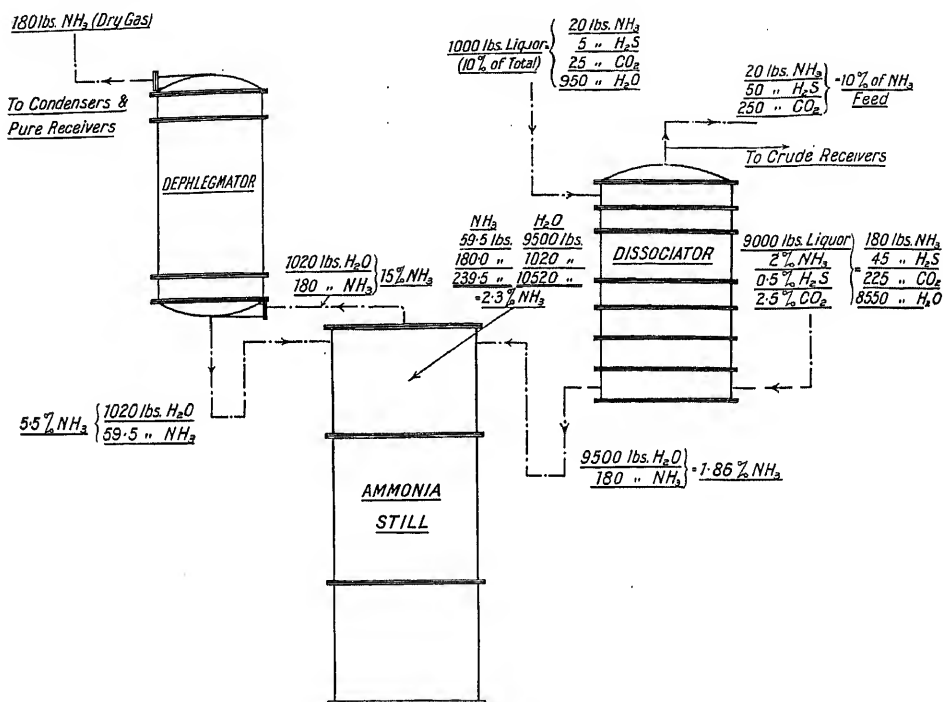


FIG. 139.—DISPOSITION OF AMMONIA AND WATER IN LIQUOR AMMONIA MANUFACTURE.

quantity of ammonia. What that quantity of ammonia will be depends on the design of the dephlegmator and its operation.

By slow and regulated cooling, and by maintaining the temperature of the steam-ammonia stream slightly below its dew-point, it is possible to aid the separation of the ammonia from the water. If the foregoing conditions are not observed, an unnecessary amount of work will be imposed on the finishing still, and the consumption of steam will be sensibly increased. This is a significant matter, and too much attention cannot be directed to this aspect, alike when designing the dephlegmator portion of the plant as in controlling its working.

It is not necessary to discuss the figures shown in fig. 139, as they are largely self-explanatory.

It will be appreciated that the figures given in the diagrams in question do not contemplate the application of steam either to the dissociator or to the finishing still. In the case of the dissociator there is no need for the application of steam, as the gas liquor has been raised to a boiling temperature coinciding with a pressure of 25 lb. per square inch above atmospheric. So far as the finishing still is concerned, the ultimate dilution of the liquor emerging from it is not under consideration. In any case, this dilution will be analogous to that prevailing in the distillation of gas liquor for sulphate manufacture.

The following figures are significant as representing the extra work imposed upon the ammonia still when producing sulphate of ammonia, concentrated ammonia liquor, and liquor ammonia :

TABLE 53

OPERATION OF A STILL WITH AN IDENTICAL STRENGTH OF GAS LIQUOR UNDER SULPHATE OF AMMONIA, CONCENTRATED AMMONIA LIQUOR, AND LIQUOR AMMONIA CONDITIONS

	Lb. of Ammonia dealt with per hour.	Lb. of Water dealt with per hour, irrespective of Dilution caused by the Introduction of Steam and Milk of Lime to the Still.
Under sulphate conditions	200	9,500
Under concentrated ammonia liquor conditions	236	10,065
Under liquor ammonia conditions (dry gaseous ammonia)	265	10,520

Where a still originally intended for use in conjunction with a sulphate of ammonia plant is utilised with other apparatus for the production of concentrated ammonia liquor, it is a wise procedure to reduce the quantity of liquor to be dealt with in the still by (say) about 15 per cent.

The important points to be observed in the working of a concentrated ammonia liquor plant may now be briefly indicated.

So far as the working and control are concerned, the plant for the manufacture of concentrated ammonia liquor may be broadly divided into two parts—one producing caustic ammonia, and the other the finishing still, with the dephlegmators and condensers, etc. It is highly important that a tolerably pure caustic ammonia should be produced if the quality of the final liquor is to be satisfactory. To produce a tolerably pure caustic ammonia, it is a distinct advantage to have a continuous record of the temperature and pressure conditions under which the dissociator is worked. This is equally applicable whether the dissociator is of the high or low pressure type. If the dissociator is of the latter type, the liquor emerging from the final tray should have a temperature corresponding to the boiling-point of water at

the particular pressure obtaining in the bottom section, otherwise the content of hydrogen sulphide in the final liquor will be above the standard.

Quite apart from the maintenance of a specific temperature and pressure in the bottom section of the dissociator, it is necessary to ensure a regular feed of liquor. This usually depends on the speed of the pumps and the pressure at which the liquor is discharged. It is an advantage to have a by-pass pipe between the delivery and suction side of the pump, on which is fixed a spring relief valve in cast iron, set at a predetermined pressure. When this pressure is exceeded, the liquor passes through the delivery to the suction side of the pump. By adopting this method, irrespective of the maintenance of a definite pressure, uniformity of feed can be ensured. Where the liquor has to be discharged through jets or sprays, it is important to ensure that the pressure does not exceed a certain limit. Increase of pressure may mean that the spray is choked.

A uniform feed of cold liquor to the dissociator for arresting any ammonia carried away with the waste gases is a matter to which frequent attention should be given. It is also necessary to use a quantity of steam to carry the waste gases to the crude receivers, or absorbers, so as to prevent sublimation occurring in the pipes.

Samples of gas liquor leaving the dissociator should be drawn continuously and tested at frequent intervals. The results of these tests will give confirmatory evidence of the satisfactory operation or otherwise of the dissociator. As regards the finishing still, etc., it will be realised that it is important to have a uniform feed of caustic ammonia from the dissociator to this apparatus. Equally, the feed of cream or milk of lime for the release of the fixed ammonia must be regular. Constancy of steam supply is equally essential. Given the latter conditions, the operation of the finishing still will be identical to what has been indicated in Chapter X. when dealing with the working and control of continuous ammonia stills.

Apart from the still, the operator will need to give attention to the working of the dephlegmators, condensers, and receivers. Assuming that the plant has been efficiently designed, and the precautions already outlined concerning the necessity for slow and regulated cooling of the steam-ammonia stream are observed, it will be found that the maintenance of a specific temperature at the outlet of the final dephlegmator (supposing two are used) will ensure the passage of such a quantity of water vapour with the ammonia as will lead to the production of the requisite strength of concentrated ammonia liquor.

Pressure gauges, in the form of U-tubes, filled with mercury and provided with a scale, should be fixed at certain points of the system between the finishing still and the final absorbers. The pressures at these various points should be taken regularly, and recorded. It is not unusual to find, due to the rapid combination or absorption of the ammonia in water in the receivers, that a vacuum is created, which may be troublesome if provision is not made for dealing with it.

At some works a vacuum breaker is fixed to each receiver. At other works a vacuum breaker is fixed to the connecting pipe between the dephlegmators and the condensers. In any case, the same purpose is served, namely, the introduction of air to satisfy the temporarily created vacuum.

Fig. 140 shows a suitable type of vacuum breaker made in cast iron, which has been found to answer satisfactorily in practice.

Constant attention should be given to the character of the discharged air from the receivers—whether crude or pure. When it is found that ammonia is escaping from the small scrubber fixed to the receivers, then steps should be taken to spray them with cold water. All receivers should be arranged in such a way as to admit of cooling when necessary.

Samples of the final liquor must be taken at regular intervals and tested, and the results recorded along with the other data indicated.

What applies to the working and control of plant when manufacturing concentrated ammonia liquor equally applies when liquor ammonia is being made.

The difference between these two operations is one essentially confined to the working of the dephlegmators and receivers. When requiring concentrated ammonia liquor, such a temperature is maintained in the outlet pipe from the dephlegmators

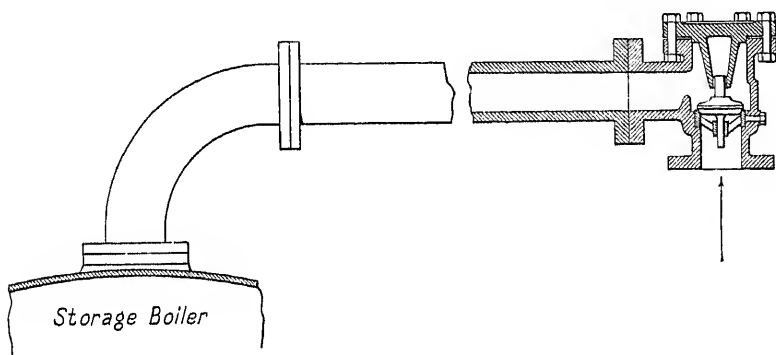


FIG. 140.—VACUUM BREAKER.

as will ensure water vapour and ammonia in such a ratio as will yield, after condensation, a product containing 25 per cent of ammonia. Where liquor ammonia is required, then a dry ammonia gas is necessary. This entails a greater volume of condensate as reflux from the dephlegmators to the finishing still. Instead of working the outlet pipe from the dephlegmators at (say) 88° to 90° C., it is necessary to work at a temperature of 20° to 25° C. The dry ammonia gas resulting may need to be freed from empyreumatic matter by passage through activated charcoal or anthracene oil, etc., etc., and subsequently will need to be absorbed in water in suitable receivers arranged to operate in series.

The work imposed on the finishing still under the respective conditions has already been enlarged upon, and it will be realised how important it is to devote strict attention to the effluent spent liquor, continuous samples of which must be drawn at regular intervals.

During the War it was found that large quantities of concentrated ammonia liquor did not satisfy the requirements of the manufacturers of ammonium nitrate, by reason of the amount of cyanogen, calculated as ammonium thiocyanate, exceeding

0.01 grm. per 100 c.c. Where a tolerably pure caustic ammonia is produced there should be little difficulty in conforming to the buyer's specification as regards cyanogen compounds. It is, however, of interest to state that the deficiency of the operation of the dissociator can be rectified by the use of powdered sulphur fed to the gas liquor undergoing dissociation. The addition of powdered sulphur is based on the fact that polysulphides are formed with sulphides of ammonium and the added sulphur. The ammonium polysulphide so formed reacts with hydrogen cyanide, producing thiocyanate. The quantity of powdered sulphur required is of the order of $\frac{1}{2}$ to 1 lb. per ton of concentrated ammonia liquor—25 per cent NH_3 —dependent on the content of volatile cyanides in the liquor. The passage of the steam-ammonia stream emerging from the finishing still through a steam jacketed purifier containing spent oxide has been used by the author with satisfactory results. The volatile cyanides were arrested as thiocyanates prior to the condensation of the steam and ammonia as concentrated ammonia liquor.

CHEMICAL CONTROL TESTS

It is not proposed to indicate other than works control tests in this connection. For more accurate work, reference should be made to the methods of analysis furnished by Linder (Alkali Inspector's Report—see Technical Index to Reports, 1894–1917), and Colman and Yoeman (*Journal of the Society of Chemical Industry*, vol. 37, Dec. 31, 1918).

The Estimation of H_2S in Concentrated Ammonia Liquor : Principle of Method

The solution is titrated with N/10 ZnCl until a drop of the liquid, filtered on to a spot of lead acetate solution as an indicator, on a white tile, just ceases to give a brown colour.

N/10 Ammoniacal ZnCl Solution

32.7 gms. of pure Zn are dissolved in pure HCl , and the solution made alkaline with NH_4OH . The volume is then made up to 1 litre. This solution is further diluted to N/10 by making 100 c.c. to 1 litre.

Lead Indicator Solution

10 gms. Rochelle salt	.	.	.	} dissolved in 100 c.c. of a 5 per cent NH_4OH solution.
10 „ NH_4Cl	.	.	.	
0.5 „ lead acetate	.	.	.	

Method of procedure for determining the H_2S content in 25 per cent concentrated ammonia liquor :

A 10 per cent solution of the liquor to be tested is made, and 10 c.c. of this solution are titrated. It is advisable to make a rough estimation in order to ascertain approximately the number of c.c. required for the titration.

$$\frac{\text{c.c. N/10 ZnCl} \times 0.17}{\text{sp. gr. of liquor}} = \text{per cent } \text{H}_2\text{S}.$$

Method employed in M.M. Laboratory

To 5 c.c. of the sample are added 15 c.c. of pure NH_4OH , diluted to 45 c.c. with water.

This is titrated in an evaporating dish with $\text{N}/5 \text{ CuSO}_4$, the contents of the basin being agitated with a stirring rod.

The point where the reaction with the H_2S is complete is marked by the blue colour of cuprammonium sulphate.

Just before this end point is reached a warning is given by the coagulation of the precipitated copper sulphide.

In cases where the H_2S content is small, 10 c.c. of the sample are taken, and 10 c.c. pure NH_4OH used.

$$\text{Per cent H}_2\text{S} = \frac{N \times 0.00341 \times 100}{Q \times \text{sp. gr.}}$$

$N = \text{c.c. N}/5 \text{ CuSO}_4$.

$Q = \text{c.c. sample taken.}$

CHAPTER XIII

SOURCES OF LOSS IN CONCENTRATED AMMONIA LIQUOR MANUFACTURE

LARGE quantities of concentrated ammonia liquor were made during the War in a variety of plants, some of which provided for the gas liquor to be preheated prior to being sent to the dissociator; others involved the pumping of the reflux to the ammonia still, and in some cases both the reflux and weak caustic ammonia leaving the dissociator had to be pumped to the ammonia still.

The gases emerging from the dissociator were dealt with in various ways. At some gas and coke oven works they were returned to the foul gas main, but almost invariably difficulty was occasioned owing to the formation of ammonium bicarbonate and/or carbamate. The deposition of an impure quality of naphthalene was not infrequent, and this tended to add to the difficulties named.

The principal sources of loss of ammonia in the manufacture of concentrated ammonia liquor may be cited as follows:

- (a) Loss from the dissociation apparatus, due to imperfect absorption of ammonia.
- (b) Loss *via* the effluent liquor from the finishing still, due to an increased volume of liquor. This loss is aggravated by imposing extra work on the stills, owing to the reflux liquor having to be dealt with.
- (c) Loss occasioned by leakage at pump glands and by spillage.
- (d) Loss owing to volatilisation of the concentrated ammonia liquor from storage boilers, due to imperfect washing of the ammoniacal air discharged.
- (e) Loss occurring in the loading of railway tanks; and
- (f) Loss arising from faulty sampling.

It is proposed to deal with these several sources of loss *seriatim*.

(a) If loss of ammonia is to be avoided in this section of the plant, it is necessary that a dissociator should be used which is designed on scientific lines. It is useless to admit partially preheated liquor at the top of the dissociator and to expect to recover all the ammonia satisfactorily. Even if ammonia is absorbed in a scrubber or wash tank, placed after the dissociator, it will be appreciated that if unnecessarily large quantities are sent forward, the gas liquor resulting will need to be distilled, and additional steam will be required, besides which, the volume of the effluent liquor will be increased. Thus the possibilities for loss are augmented at several points.

The plant required for the desulphurisation and decarbonation of gas liquor can be divided broadly into four parts: (1) that assigned for the application of heat to the liquor, either by the use of direct steam or products of combustion, or by partially preheating (interchange heat), and the subsequent employment of steam or products of combustion; (2) that required for the cooling of the gases released in the dissociation, so as to aid the absorption of the ammonia not held in solution; (3) that needed for the absorption by gas liquor of the ammonia referred to in (2);

and (4) that required for the removal of the traces of ammonia escaping the absorption plant mentioned under (3), by water. A drawing of such plant is given in fig. 141, and it need hardly be said that such an arrangement is flexible in the matter of control, and calculated to reduce the loss of ammonia to a minimum.

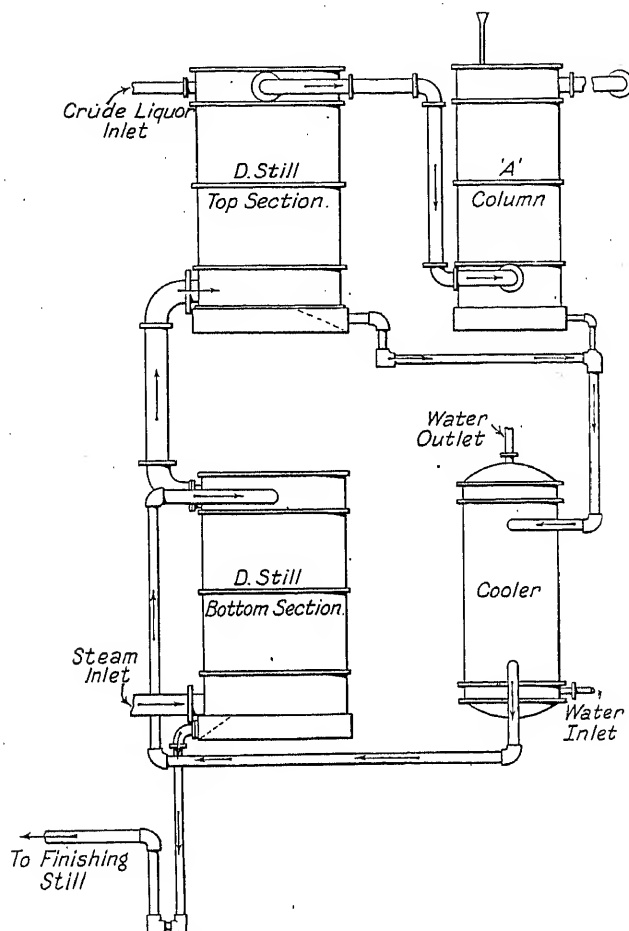


FIG. 141.—DISSOCIATOR PLANT EMBRACING APPARATUS ASSIGNED FOR EACH FUNCTION.

It will be seen that the gas liquor used for the absorption of the ammonia not held in solution is cooled by water in a multitubular cooler prior to being sent to the dissociation section proper. In this way the temperature of the gases leaving the decomposing section of the plant can be better controlled than if an attempt were made to cool the gases themselves by water.

Of course other combinations of the plant are possible, as has been indicated in Chapter XII. But all embody the four broad principles which have been elaborated above.

The extent to which loss of ammonia will occur will depend on the efficiency or otherwise of the plant and its operation. One of the chief sources of loss in connection with the decomposing plant lies in the failure to arrest the ammonia associated with the spent gases.

It is not generally appreciated that the absorption of ammonia from the effluent gases in question is a different proposition from that of the absorption of ammonia from crude coal gas.

A comparison of the respective analyses of the two gaseous mixtures will make this evident. The table below shows the impurities in condensed but unwashed gases. Most of the particulars are taken from figures furnished by Lewis T. Wright.

TABLE 54

Class of Coal.	CO ₂ per cent, by Volume.	H ₂ S per cent, by Volume.	NH ₃ per cent, by Volume. (Calculated.)	Equivalent True Coal Gas. Per cent by Volume.
Newcastle	1.5	1.4
Yorkshire silkstone . . .	1.5	1.3	1.25	(say) 96.0
Derbyshire	1.5 to 2.3	1.0 to 2.0

Analyses taken of the effluent gases leaving a decomposer, the working of which the author had occasion to investigate some years ago, were as follows :

TABLE 55

	Per cent.
Air	5.0
NH ₃	15.5 (Outlet temperature of gases
H ₂ S	9.2 from decomposer, 46° C.)
CO ₂	69.4

99.0

Reference to the table relating to the impurities in condensed but unwashed gas shows that such impurities are relatively small. Undoubtedly, the gases constituting coal gas exert a carrying influence, and either obviate a reaction between the ammonia and carbon dioxide at the temperature obtaining, or otherwise carry forward by entrainment any ammonium carbonate which may be formed. A different condition prevails with the effluent gases from a decomposer. Owing to the high concentration of carbon dioxide and ammonia, opportunity is afforded for reaction. The result is that under favourable conditions of temperature ammonium bicarbonate and other

complex compounds of ammonia are formed as a sublimate in the pipes leading to the absorber, or in the gas mains used for conveying the foul gases to the plant assigned for dealing with the hydrogen sulphide content.

The mixture of gases represented in Table 55 is a peculiarly insidious one, and if any escaping ammonia is to be arrested satisfactorily it is important that the scrubber, or wash tanks, should be contiguous to the dissociator, and that the pipe conveying the effluent gases should not be exposed to unfavourable atmospheric conditions. It has been found in practice that the use of a small quantity of steam, provided the temperature of the gases is not unduly raised, as a carrying medium, is an advantage. Such steam prevents the deposition of a sublimate prior to the gases reaching the catch absorber. This latter should preferably take the form of a boiler (or series of boilers), of tolerably large dimensions, say, 5 ft. diameter by 20 ft. long, for a plant dealing with 60 to 80 thousand gallons of liquor per day. The boilers in question should contain water, and the effluent gases should be introduced to them by a suitable perforated distributing pipe or pipes.

If the foregoing points are observed, and the precautions indicated are acted upon, loss of ammonia from this source can be reduced to a minimum.

(b) In the working of most concentrated ammonia liquor plants it is correct to say that the bulk of the effluent liquor is much greater than obtains when the manufacture of sulphate of ammonia is being undertaken. Moreover, the ammonia content of such liquor is usually in excess of the standard adhered to when sulphate of ammonia is being manufactured. Improvement can only come from the employment of an efficient type of dissociator, and from a greater appreciation of the principles involved in dephlegmation. Invariably the content of ammonia returned to the still *via* the reflux liquor is needlessly high, and the imposition of additional work on the still invariably leads to a high ammonia content in the effluent liquor.

(c) Pumping should be avoided as far as possible, in order to reduce loss in this connection. By working the dissociator under pressure it is possible to feed the ammonia still with a uniform quantity of weak caustic ammonia without having recourse to pumping.

Where pumping is necessary, it will be found that the use of a deeper stuffing box to afford extra packing, or the adoption of a pump with multiple glands, will reduce loss from this source.

(d) It is not generally appreciated how serious is the loss from storage boilers, due to the action of the sun's rays, and to loss *via* the vented air during filling. It is preferable to place all boilers in a covered building, so as to obviate unnecessary surface heating. Where this is impracticable, then in the summer months it is wise to keep the tanks cool by using a spray of cold water. All vented air should be washed, and the washings sent to the gas liquor storage tanks or wells.

(e) To avoid loss in the loading of railway tanks, these should be fitted with loading pipes carried to within 3 in. of the bottom of the tank, so as to avoid undue agitation, and the tank should be sampled, when filled, through the smallest size hole cut in the manlid, compatible with securing a representative sample. In summer

the tanks should be sprayed with cold water before opening the miniature manlid named.

(f) Dip samples taken by a long glass rod, or a long iron pipe with a small bottom valve, operated by a screw-spindle from above, should be discountenanced. While by such means it is possible to get a representative sample of the concentrated ammonia throughout the full depth of the tank, it will be found that the liquor adhering on the outside of the tube loses ammonia rapidly by volatilisation, and as this liquor flows into the sample bottle, which is filled by means of the glass rod or iron tube, the sample is not representative of the bulk of the concentrated ammonia liquor in the tank. In point of fact, the ammonia content of the sample will be less than that of the bulk. The best method to adopt is to dip the sample bottle itself into the tank. The neck of the sample bottle should be of a definite size so as to admit of the steady introduction of the liquor. In other words, the displacement of air from the sample bottle must be such that a representative sample throughout the depth of the tank is secured. In this way, if there is any stratified layer of concentrated ammonia liquor, the sample is likely to reflect such stratification. On withdrawal from the tank, the outside of the sample bottle should be dried. Subsequently the bottle should be hermetically sealed.

With a highly volatile liquid such as concentrated ammonia liquor, it will be appreciated that attention to many minute details such as have been indicated, is essential, if a high over-all yield is to be ensured.

CHAPTER XIV

TREATMENT OF THE WASTE GASES

IN the distillation of gas liquor in the manufacture of sulphate of ammonia, hydrogen sulphide, carbon dioxide, and hydrocyanic acid, along with pyridine, hydrocarbons, and steam, pass unabsorbed from the saturator to the condensing plant. These gases, as has been explained in Chapter VII., are cooled initially by gas liquor, and subsequently by water.

The devil liquor resulting is sent to a sectional plated cast-iron tank, and is pumped along with the gas liquor to the distillation plant. Some of the properties of this liquor will be dealt with in the next chapter.

It is important that the noxious gases named should be cooled to atmospheric temperature, so as to reduce the quantity of water vapour entrained to a minimum. In the dissociation of gas liquor for the production of weak caustic ammonia, in the manufacture of concentrated ammonia liquor or liquor ammonia, the noxious gases—hydrogen sulphide, carbon dioxide, and hydrocyanic acid, along with the other impurities previously named, usually pass to an absorption tower, to ensure the complete elimination of any unabsorbed ammonia. They are then available for treatment.

METHODS OF TREATMENT, AND THEIR AVAILABILITY

Recourse is generally had to one or other of four methods of treatment, the adoption of which depends largely on local considerations. These methods may be cited as follows: (1) The conversion of the H_2S into SO_2 , which is used in the manufacture of sulphuric acid; (2) the combustion of H_2S to SO_2 , and the absorption by calcium carbonate; (3) the conversion of H_2S into sulphur by the Claus process, and (4) the absorption of H_2S by passage through oxide of iron. The author has had occasion to operate all four methods.

The first method is essentially one for a large works where the manufacture of sulphuric acid is conducted. The second method is available for small or medium-sized works where the size of the plant does not warrant the manufacture of sulphuric acid, and where it is not desired to operate the Claus process or to use oxide of iron. The third method is unsuitable for small works where chemical supervision is absent, but is satisfactory for medium-sized and large works producing the equivalent of 5 tons or more of ammonium product—25 per cent NH_3 —per day. The fourth method is alike available for any size of works.

1. THE CONVERSION OF HYDROGEN SULPHIDE INTO SULPHUR DIOXIDE

The importance of cooling the waste gases to atmospheric temperature has already been emphasised. This is essential if they are to be burned for the production of sulphur dioxide. Provision should be made in the foul gas main leading from the coolers of the sulphate of ammonia or the final absorption tower of the concentrated

ammonia liquor plant, for the withdrawal of any water vapour that may be mechanically entrained, prior to passing the gases forward to the burners. It is advisable to fix one or two baffle boxes at certain sections of the main in question, in order to facilitate the removal of water vapour and any liquid hydrocarbons mechanically carried with the waste gases. The type of baffle box suggested is shown in fig. 142.

It is seen that provision is made for continuously "draining-off" the arrested liquid products to a seal pot. The liquor overflowing the latter can with advantage be collected, as it will be found to contain appreciable quantities of crude pyridine, which can be separated from the associated water in a suitable conical separator, and sold to the local tar distillery at a remunerative price.

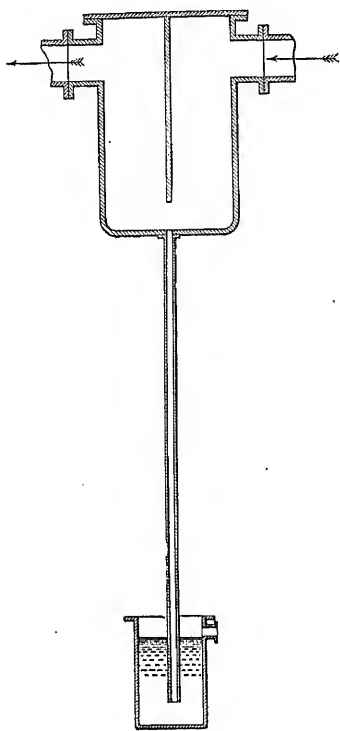


FIG. 142.
BAFFLE BOX FOR WASTE GASES.

Where the production of sulphuric acid is also carried out, the combustion of the waste gases is invariably effected in spent oxide or pyrites burners. At some works, where mechanical spent oxide burners are in operation, it is usual to assign a small section of the old hand-operated spent oxide burners for this purpose, and merely to burn the lump spent oxide, which is unsuitable for mechanical burners. In this way crushing of the spent oxide lumps is avoided, and the combustion of the latter in the hand burners affords the necessary heat for maintaining a satisfactory combustion temperature.

Fig. 143 shows an elevation and section of a set of spent oxide burners dealing with the waste gases from a sulphate of ammonia plant producing 20 tons of sulphate of ammonia per day. On this set 9.6 tons of spent oxide are burned in addition to the waste gases.

It will be seen that there are five tiers of shelves, the top one of which is reserved for the combustion of the hydrogen sulphide of the waste gases. This particular shelf intervenes the fourth shelf and the top main flue. Thus, at all times there is a sufficiently high temperature to admit of the ignition of the hydrogen sulphide when it meets with excess air supplied *via* the oxide shelves. The air for combustion is admitted under suction by the operation of a fan at the end of the plant, through the apertures in the doors of those shelves on which the spent oxide is charged.

It will be seen that easy means are afforded for cleaning the 6-in. cast-iron main, as also the several 2-in. wrought-iron tubes through which the gases are passed to their individual combustion chambers. Fifteen 2-in. pipes suffice to deal with the whole of the waste gases resulting from the operation of the sulphate of ammonia plant in question.

It is estimated that 250,000 cubic feet of gas are dealt with per day. The hydrogen sulphide content varies between 12 and 15 per cent.

It is important to keep the main and the several 2-in. wrought-iron tubes free from deposited tarry matter. These should be cleaned at least every fourteen days. An aid to speedy cleaning is to be found in soaking a little cotton waste with paraffin, and igniting it in the mains, and in the openings provided by the several wrought-iron tubes. Afterwards an auger and rake should be freely used for withdrawing the soft tarry deposit. After cleaning, the wooden plugs are driven home tight.

The combustion of the waste gases in spent oxide burners undoubtedly affords a ready and inexpensive means of treatment. The only disability attending this method is that diluent gases (largely carbon dioxide) are sent forward to the sulphuric acid chamber plant, and these occupy valuable space intended for reaction. Where the proportion of waste gases burned in relation to the spent oxide combusted is low, no difficulty will be encountered from the high ratio of water resulting on combustion of the sulphuretted hydrogen, but this may constitute a disadvantage,

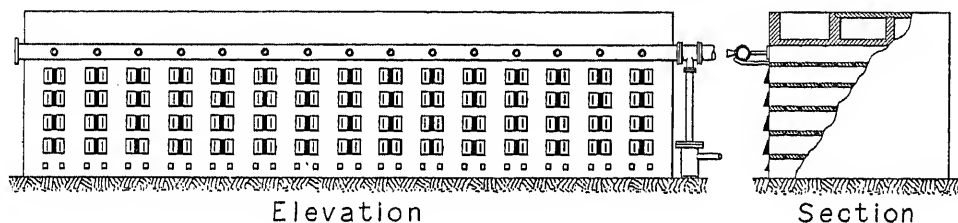


FIG. 143.—SPENT OXIDE BURNERS FOR THE COMBUSTION OF H_2S .

by reason of the production of weak acid, in connection with those sulphuric acid plants where the waste gases in question are the exclusive source of sulphur.

It must not be assumed from the foregoing that it is impossible to manufacture sulphuric acid by the exclusive use of the H_2S of the waste gases. This has often been done. Indeed, reference to the 36th Annual Report on Alkali, etc., works will reveal that the district inspector of the eastern and south-eastern counties—F. Napier Sutton—reported as follows: "Sulphuretted hydrogen as a source of sulphur for acid making is in use at six works, and a very large quantity of acid is now produced from this noxious waste product. Further trial has been made to work a set of chambers with sulphuretted hydrogen alone at a works at which the gas is available in large quantity, and a trial of several months' duration has been very successful. SH_2 to the equivalent of 3.75 tons of sulphur per twenty-four hours has been passed through the burners to the chambers, which have been worked at a capacity of 17 cubic feet per lb. of sulphur burned per twenty-four hours. In all other cases the sulphuretted hydrogen is burned either in pyrites or spent oxide kilns in conjunction with these materials."

Apart from the ready means which the above method affords for dealing with noxious gases, their combustion in spent oxide burners enables one to deal with low-grade spent oxide, the satisfactory treatment of which would be impossible but for the extra temperature resulting from the burning of the waste gases.

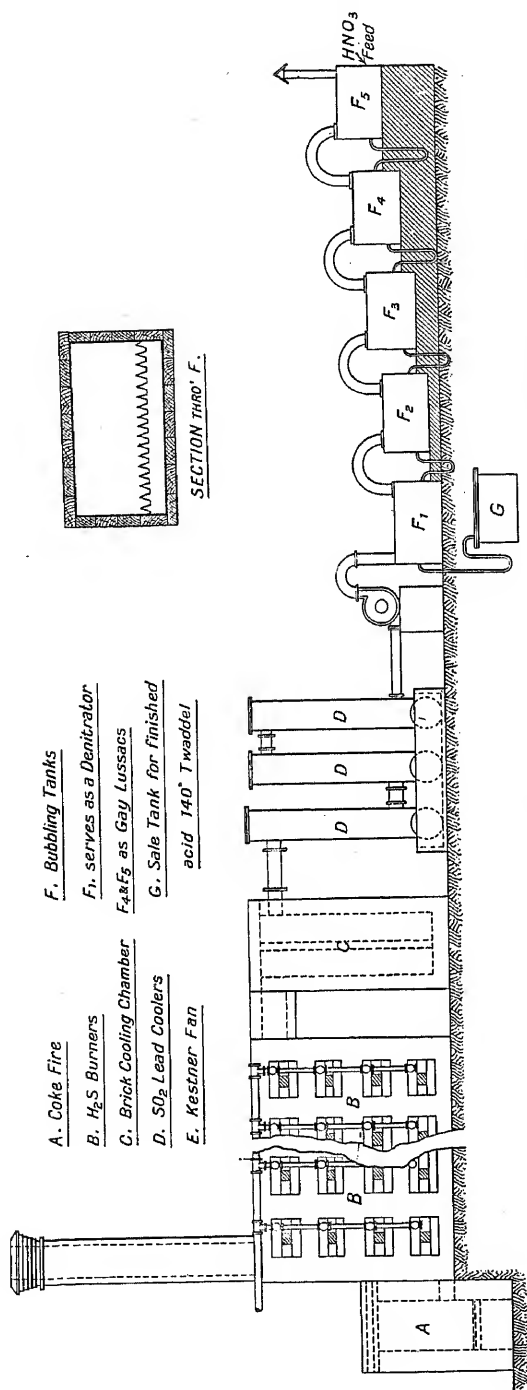


FIG. 144.—MODERN PLANT FOR THE MANUFACTURE OF SULPHURIC ACID FROM SATURATOR GASES.

It is important, however, to avoid the escape of the slightest trace of ammonia from the saturator, otherwise an adverse influence on the operation of the chamber plant will be established. Nothing is calculated to disturb the normal operation of the acid manufacturing process more than the passage of traces of ammonia with the waste gases. This prejudicial effect is very rapid.

The author recently inspected plants at two large continental works where the waste gases were being burned, and the products of combustion discharged via a chimney-stack. At one works 40 tons of sulphate of ammonia were being produced per day. Taking the hydrogen sulphide content of the gas liquor at 0.3 per cent in relation to the ammonia at 1.8 per cent, it will be seen that $\frac{40}{1} \times \frac{25.5}{100} \times \frac{3}{10} \times \frac{10}{18} = 1.70$ tons of sulphur were being combusted in the above manner per day.

The above approximates to 15 per cent of the sulphuric acid requirements of the sulphate of ammonia plant. The sulphur dioxide resulting from the combustion of the waste gases in question, based on a 90 per cent efficiency, would produce $1.70 \times 3.93 = 6.68$ tons of 70 per cent sulphuric acid per day.

The Manufacture of Sulphuric Acid from Waste Gases — A Modern Plant

There are one or two peculiarly simple forms of sulphuric acid plant which can be advantageously adopted for the manufacture of sulphuric acid from the SO_2 resulting from the combustion of waste gases. Fig. 144 shows such a plant. It will be seen that the

arrangement comprises a burner for the combustion of the hydrogen sulphide of the waste gases, this burner being arranged in such a way that extraneous heat from the products of combustion from a coke fire can be applied, should there be any diminution of temperature at any time in the self-combustion process. This heating device is also available for starting up the burner plant, which must be initially heated to the ignition point of the waste gases before passing the latter forward for combustion.

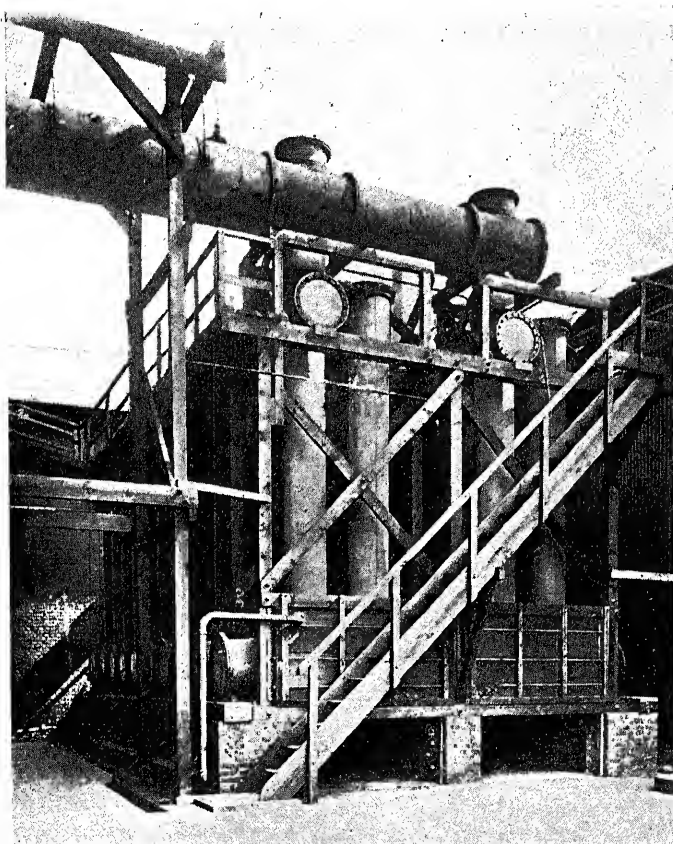


FIG. 145.—SO₂ COOLER.

The burner gases are subsequently partially cooled in specially designed and operated SO₂ coolers, a large installation of which is shown in fig. 145. A Kestner fan is introduced between the coolers and the "bubbling tanks." Thus the necessary air for combustion of the waste gases is introduced to the burners under suction, and the partially cooled burner gases are passed under positive pressure to the bubbling tanks, which contain nitrosulphonic acid of a well-defined strength. By reason of the intimacy of contact between the sprayed nitrous acid (which is main-

tained in excess) and the sulphur dioxide, rapid reaction occurs with the formation of sulphuric acid. Water can be admitted to the bubbling tanks as desired.

A Glover tower may be employed, or one of the bubbling boxes may function as a Glover tower. Similarly, one or more Gay-Lussac towers of the usual design may be employed, or the bubbling boxes may serve as absorbers of the oxides of nitrogen.

This arrangement of plant is inexpensive, yet withal flexible and foolproof in operation. It can be worked at the phenomenally low capacity of less than 1 cubic foot of chamber space per lb. of sulphur per twenty-four hours. The only matter of importance is to ensure the presence of an excess of nitrosulphonic acid. These bubbling tanks are the subject of a patent, No. E.P. 156328, of 1919.

Where it is desired to produce sulphuric acid at a low cost from waste gases such as those arising from sulphate of ammonia plants, or from copper smelting or other such metallurgical works, the plant described is peculiarly satisfactory.

2. THE COMBUSTION OF H_2S TO SO_2 , AND ABSORPTION BY CALCIUM CARBONATE

At one or two works in the South of England, the waste gases are led by a pipe over a small coke fire, and the resulting burner gases are passed up towers, down which the supernatant spent ammonia liquor is irrigated. In this way decoloration of the effluent liquor is secured, and this appears to satisfy the local authorities. Other works burn the H_2S of the saturator gases to SO_2 in a suitable furnace, such as is indicated in fig. 146. A gas jet, or coke fire is used for securing incipient combustion, and for the maintenance of such a temperature as will ensure continuity of the operation. Provision is made in the burner for the admission of a secondary supply of heated air in order to burn

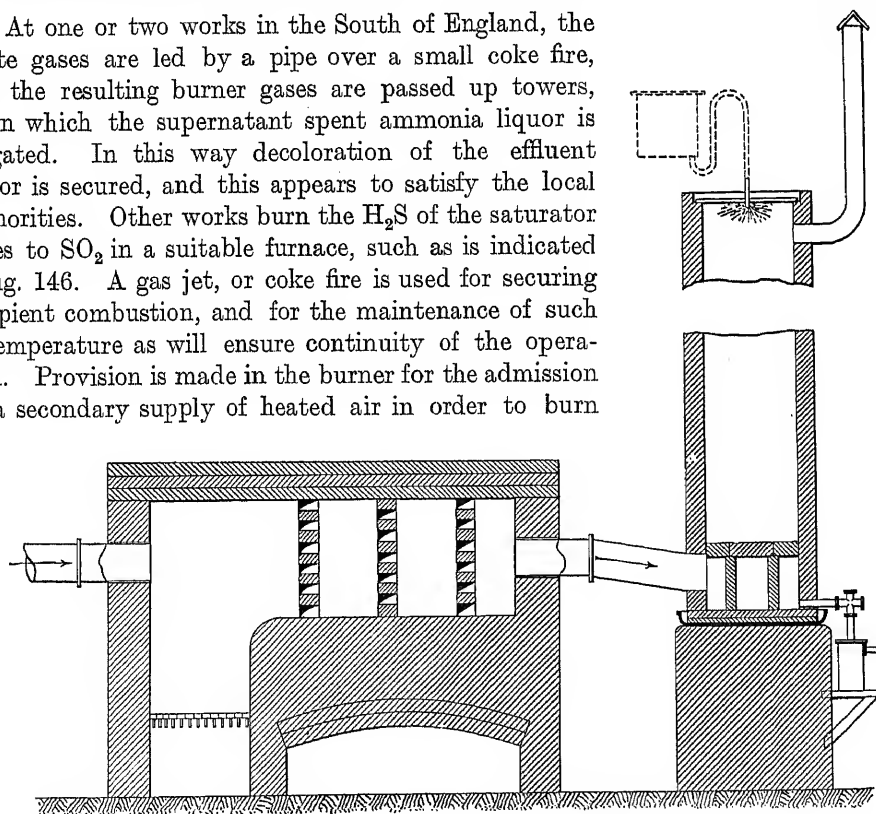
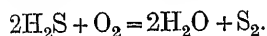


FIG. 146.—PLANT FOR THE COMBUSTION OF H_2S TO SO_2 .

any sublimed sulphur to sulphur dioxide. After ignition, the gases are passed through several series of chequerwork walls, so as to prevent any sulphur vapour passing away unoxidised. This is a matter of supreme importance, otherwise not only will the sulphur deposits cause dislocation of the operation of the absorbing section of the plant, but absence of draught, caused by the choking of the tower, will result in a deficiency of air, which will give rise to further quantities of sulphur vapour. It will be appreciated, therefore, that there must be a good command of draught at the furnace. Apart from this, the resulting gases containing SO_2 must be cooled to a temperature of about 25°C . if satisfactory absorption is to take place. This method has proved quite satisfactory at several works throughout the country, but of course it involves expense, and there is no revenue from any resulting product.

3. CONVERSION OF THE H_2S OF THE WASTE GASES TO SULPHUR—CLAUS PROCESS

This process consists in mixing the waste gases containing hydrogen sulphide with such a quantity of air as will produce the reaction indicated below, when the gaseous mixture is passed through the red-hot catalytic medium provided.



Arrangement of the Claus Plant

The usual arrangement of Claus sulphur recovery plant, supplied by C. & W. Walker, Ltd., is shown in fig. 147. It comprises : (a) A large cast-iron cylinder, lined

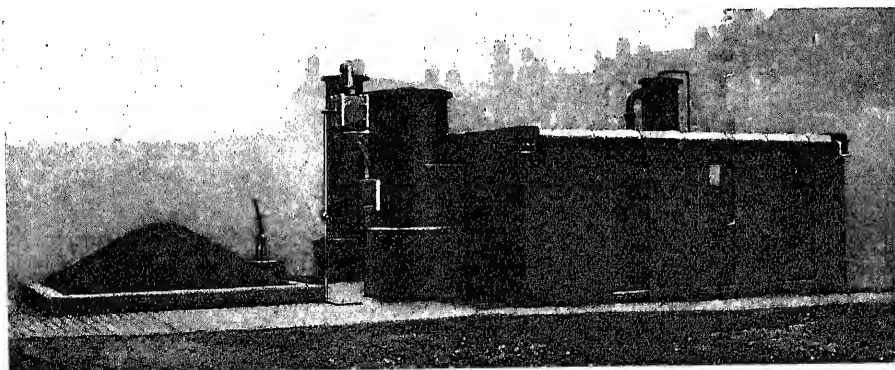


FIG. 147.—CLAUS KILN.

with firebricks and having a firebrick grate, on which rest loose firebricks, and over these is a layer of hydrated oxide of iron. Sometimes pyrites cinders of suitable size are used as the catalytic medium. (b) A cast-iron air box attached to the upper portion of the kiln, in which the air and foul gases mix before entering the kiln. (c) An air engine with automatic blow-off valve, to give a regular and continuous supply of

air. (d) A subliming chamber provided with transverse baffles to aid deposition of the finely divided sulphur. In this case the chamber is built of ordinary brickwork, lined at the portion nearest the kiln with firebrick. (e) A scrubber filled with limestone, down which a small stream of water is constantly flowing. The scrubber serves the purpose of arresting the sulphurous acid gases, which invariably emerge in greater or lesser quantity from the depositing chamber; and (f) an open purifier, filled with oxide of iron, for absorbing any hydrogen sulphide which may escape.

Method of Operation of the Claus Sulphur Recovery Plant

The brickwork of the grate is heated to a dull red heat prior to commencing the operation of the Claus kiln.

The waste gases leaving the saturator are first cooled by liquor, and afterwards by water, to atmospheric temperature, as has been explained. Subsequently they are conducted into the air inlet box and are admixed with the air from the air engine, or compressed air supply, as the case may be. The gaseous mixture enters the kiln at the top and passes downwards through a layer of hydrated oxide of iron, when reaction is set up. Although the production of sulphur has been represented by a simple equation, it should be remarked that more reactions than one take place, and that they are of a somewhat complicated character. Reference to this aspect will be made later.

Due to the rapidity of the reactions in the formation of sulphur, the complete mass of oxide soon attains an incandescent state. When this temperature is reached the sulphur produced is vaporised, and passes with the other associated gases through the firebrick grating at the bottom of the kiln into the subliming chamber, where the sulphur vapours are cooled and deposited. The condensation of the water, usually in the form of a spray, is effected in the subliming chamber, from which it drains—preferably *via* sealed pipes. The uncondensed gases emerging from the subliming chamber enter a wet limestone scrubber, and thence pass through an oxide of iron purifier, finally emerging devoid of all noxious constituents.

An excess of air will promote the formation of sulphur dioxide, which is arrested in the limestone tower, whereas a deficiency of air will result in hydrogen sulphide as such passing forward from the subliming chamber, and the function of the oxide of iron purifier is to arrest effectually this gas.

The efficiency of recovery varies between 80 and 90 per cent, according to the extent of the supervision exercised. At some works the process is conducted without any attention over and above that which is available in the manufacture of sulphate of ammonia.

Dimensions of a Claus Sulphur Plant

Some idea of the size of a Claus plant is furnished in the Alkali Inspector's Report, No. 21, p. 81, where a description is given of the plant installed at the Leicester Corporation Gas Works for the treatment of the saturator gases arising from a weekly production of 35 to 40 tons of sulphate of ammonia. A plan of the essential parts of the plant in question is shown in fig. 148. The kiln A

comprises an iron shell, with 18-in. brick lining, 3 ft. wide by 5 ft. long inside. A perforated partition *c* causes an intimate mixture of the gases and air entering at *a* and *b*. The inner chamber, with an area of 3 ft. by 3 ft., is filled with lumps of oxide of iron containing a little manganese 1 in. by $1\frac{1}{4}$ in. diameter. In chamber B, which is divided by transverse walls, as shown, the finely divided sulphur is deposited.

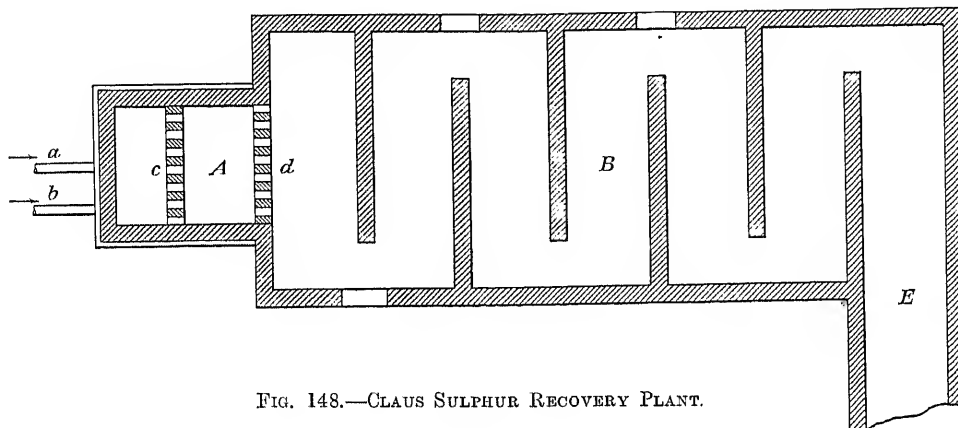


FIG. 148.—CLAUS SULPHUR RECOVERY PLANT.

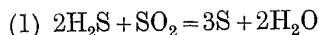
From B the effluent gases traverse a flue 2 ft. 6 in. wide by 58 yd. long, in which are built a number of baffle walls. At the end of the flue, which takes the form of a hollow square, is disposed the scrubber. This is 24 ft. high by 4 ft. diameter, packed with limestone, down which water is percolated. The SO_2 is here arrested. Any remaining H_2S is absorbed by the open oxide of iron purifier, 12 ft. 10 in. square.

Owing to the difficulty of regulating the process to a nicety, it is essential that a limestone tower and purifier should be employed. It is known that acidities as high as 12 grains of SO_3 per cubic foot have been recorded on Claus sulphur plants.

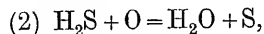
Reactions

Carpenter and Claus have made a detailed investigation of the reactions in the Claus process. Particulars will be found in the *Journal of the Society of Chemical Industry*, 1904, p. 557, *et seq.*

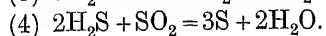
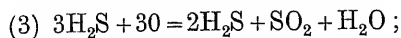
In addition to the well-known reactions



and



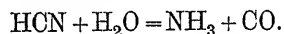
evidence is available which indicates that the following reactions occur :



On the basis of many experiments, not alone conducted on the laboratory scale, but of a technical nature, and from observations made at several works, attempts

were made by Carpenter and Claus to determine the conditions prevailing in the kilns, the factors governing the yield of sulphur, and the rôle played by ammonia.

Ammonium sulphite and sulphate were not infrequently found, but ammonium sulphide was never detected. The presence of ammonium sulphite can be easily explained, as sulphur dioxide is always present in greater or lesser quantity in the gases leaving the catalyst, and there must be times when a trace of ammonia is by-passing the saturator, due to the formation of rock salt, or to low acidities of the bath liquor. Moreover, it must be remembered that hydrocyanic acid undergoes hydrolysis, with the formation of ammonia, thus :



Most samples of recovered sulphur from Claus kilns contain appreciable quantities of ammonium salts, and it is necessary that these should be washed preferably in a centrifugal before sending the material to the acid works for combustion. Ammonium compounds as high as 12 per cent, estimated as ammonium sulphate, have been found present in the recovered sulphur withdrawn from Claus kilns.

4. ABSORPTION OF HYDROGEN SULPHIDE BY PASSAGE THROUGH OXIDE OF IRON

The use of oxide of iron is usually resorted to at small works for the absorption of H_2S from saturator gases. Thirty years ago it was generally customary to use cast-iron boxes of the ordinary gas works type, or brick boxes. In many cases these were open at the top. Both these types of purifier were provided with grids, and the gases were passed in an upward direction. In some cases only one tier of oxide was used. In others it was the practice to use multiple tiers. When sulphiding became visible, the box was thrown out of action and another one started. In some cases air was introduced to the spent box by means of an air injector, so as to aid revivification. In other cases the material was removed from the purifier and spread in an even layer on a suitable floor, so as to induce oxidation of the ferrous and ferric sulphides to ferrous and ferric oxides.

About twenty years ago it was suggested that the oxide of iron should be arranged in the form of a conical heap on a concrete floor, the foul gases being introduced thereunder by a simple gas-distributing device. This method rapidly gained in popularity, and is now generally practised at most small sulphate of ammonia works. Fouling is very easily detected, less labour is entailed in operation, and larger surfaces are available for oxidation.

A particularly suitable hydraulic change valve for use when changing from one purifier to another is shown in fig. 149. This type of valve is made by C. & W. Walker, Ltd., of Donnington (Salop).

Fig. 150 shows the lay-out of a purifying system capable of dealing with the saturator gases from a plant producing seven tons of sulphate of ammonia per day.

It will be seen that the heap system is the one adopted. After cooling to atmospheric temperature, the waste gases are passed along an 8-in. pipe, thence through four 6-in. pipes fixed at right angles, on each of which a valve is placed. It is usual

to work two heaps on and two off. The gases emerge from the 6-in. pipe, on which is placed an iron plate, and are then distributed laterally through the openings A, A, A, A into a small square structure B. A 7 ft. square concrete wall, 12 in. high by 6 in. thick, along with a smaller square structure B, serves to carry the grids on which the oxide of iron is placed. At some works virgin oxide is not used, but it is preferred to work up material containing about 30 to 35 per cent of sulphur, arising from the purification section of the carburetted water gas plant.

In starting such heaps, the space occupied by each is indicated by X, X, X, which represents roughly 300 cub. ft., and involves the use of about 10 tons of oxide of iron. Two such heaps are put into commission, and will suffice for the absorption of the H_2S of the saturator gases for about ten to fourteen days without disturbance, but with the addition of small quantities of material added daily to cover that portion of the heap which has revealed signs of sulphiding. After the elapse of ten to fourteen days it is necessary to stop the passage of the gases, and to turn on to the two other freshly prepared heaps.

Meanwhile, it will be found that the spent heaps to which additional quantities of oxide of iron have been added daily have reached the dimensions shown by Y, Y, Y, representing about 800 cub. ft. of material. It will be appreciated that the oxide increases in weight by reason of the accumulation of sulphur.

The spent heaps are then broken down, and the material is spread over a concrete floor in a layer of 8 to 12 in. in depth. The material remains in this position, undergoing revivification about every ten days, after which it is necessary to prepare fresh heaps to be available when those at present at work give evidences of becoming foul. The back pressure, in terms of water gauge, by this method need not exceed 1 in. The heaps can be attended to by the man responsible for the preparation of the milk of lime.

The cost of this method of purification, from the point of view of labour, varies from 1s. to 1s. 6d. per ton, according to local circumstances, and the extent to which the materials have to be carted.

Reference to the Alkali Inspector's Report, No. 31, shows that the cost of purification of the saturator gases by oxide of iron varies in seven cases from 7s. 1d. to 1s. 6d. per ton of sulphate of ammonia, and in the case of five works operating the Claus

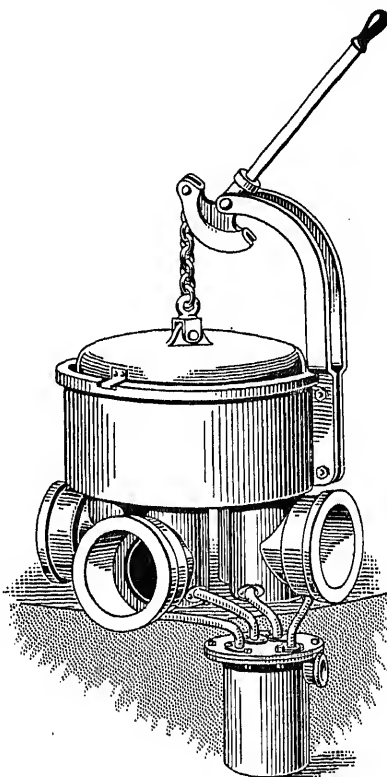


FIG. 149.—HYDRAULIC VALVE.

sulphur process the cost of purification varies from 1s. 1½d. to 8d. per ton of sulphate of ammonia. In no case was there any actual profit.

With a good quality of bog ore, or artificial oxide of iron, it is possible for the

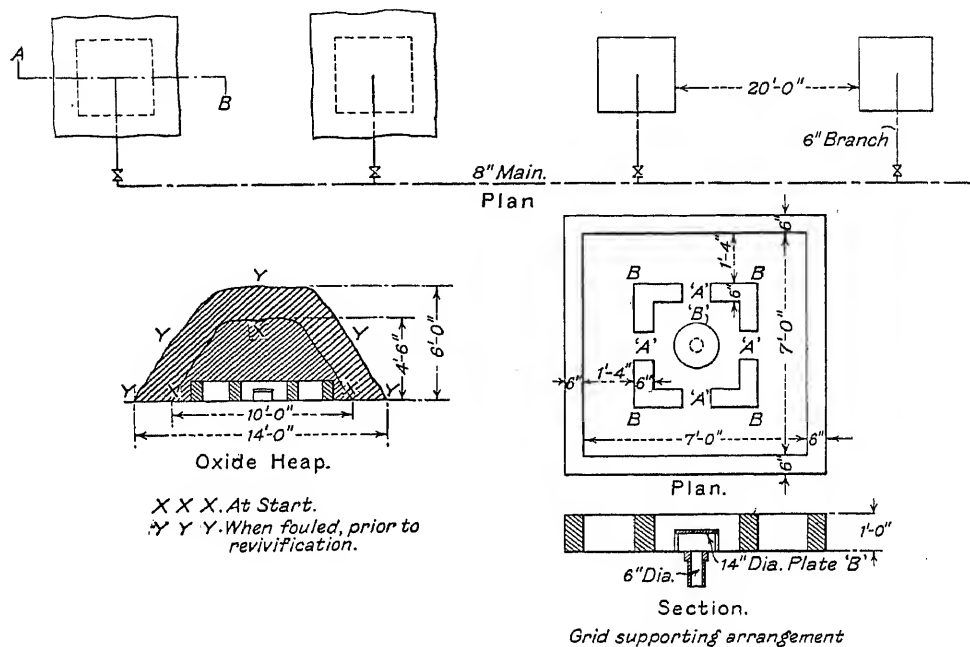


FIG. 150.—GENERAL LAY-OUT, ETC., OF PURIFICATION PLANT.

oxide to accumulate 60 per cent of free sulphur in the purifiers attached to a sulphate of ammonia plant.

It is a much easier proposition to purify gases having a concentration of H_2S varying from 13 to 20 per cent by volume, than to purify crude coal gas which contains only 1.3 to 1.5 per cent of H_2S by volume.

CHAPTER XV

THE TREATMENT OF EFFLUENT LIQUOR AND THE DISPOSAL OF THE SLUDGE

THE problem of the treatment and the disposal of the spent liquor discharged from ammonia stills becomes increasingly complex and difficult.

River authorities are much more vigilant than was the case a decade or two ago, and they are not now inclined to accept, as was their wont, spent liquors of a noxious character, if the volume is such as is calculated to cause trouble at the sewage works.

By the Pollution of Rivers Act of 1876, County Councils have power to restrain a person or firm from polluting a stream, but if that person or firm can show to the satisfaction of the court "that the best practicable and reasonably available means to render harmless the poisonous, noxious, or polluting liquid so falling or flowing or carried into the stream" have been employed, then no offence shall be deemed to have been committed.

Again, provisions are contained in the Public Health Acts, Amendment Act 1890, Part 3, Clause 17, Sections 1 to 3, which read as follows :

1. Every person who turns or permits to enter into any sewer of a local authority or any drain communicating therewith—
 - (a) Any chemical refuse, or
 - (b) Any waste steam, condensing water, heated water, or other liquid (such water or other liquid being of a higher temperature than one hundred and ten degrees of Fahrenheit),which, either alone or in combination with the sewage, causes a nuisance or is dangerous or injurious to health, shall be liable to a penalty not exceeding ten pounds, and to a daily penalty not exceeding five pounds.
2. The local authority, by any of their officers either generally or specially authorised in that behalf in writing, may enter any premises for the purposes of examining whether the provisions of this section are being contravened, and if such entry be refused, any justice, on complaint on oath by such officer, made after reasonable notice in writing of such intended complaint has been given to the person having custody of the premises, may by order under his hand require such person to admit the officer into the premises, and if it be found that any offence under this section has been or is being committed in respect of the premises, the order shall continue in force until the offence shall have ceased or the work necessary to prevent the recurrence thereof shall have been executed.
3. A person shall not be liable to a penalty for an offence against this section until the local authority have given him notice of the provisions of this section, nor for an offence committed before the expiration of seven days from the service of such notice, provided that the local authority shall not be required to give the same person notice more than once.

It will be appreciated that three considerations are of significance. These are (a) the volume of the spent liquor discharged to the sewer in relation to the volume of the sewage, (b) the nature of the sewage, and (c) the best practicable and reasonably available means to render harmless the noxious liquid.

Where the volume of the spent liquor does not exceed 0.5 per cent of the total sewage, exception is rarely taken to its discharge.

Where the sewage is of an acid nature, due to pickling liquors from metal works, or from factories using acid, it is not improbable that the authorities will welcome not only the spent liquor, but will allow the discharge of the lime sludge with it. In this case it is only necessary to cool the spent liquor below the specified temperature.

As to what constitutes the best practicable and reasonably available means to render the noxious spent liquor harmless is a matter which will be the subject of detailed consideration later.

COOLING THE SPENT LIQUOR

The first consideration which should occupy the technician is the question of the cooling of the effluent liquor. Is it intended to take advantage of the potential heat of the discharged liquor, and thus conserve heat units, or must some plant be erected which involves the use of water or air as a cooling medium? Obviously, in the interests of economy, advantage should be taken of the potential heat of the spent liquor.

The author has already referred to a peculiarly satisfactory method of heat interchange (see Chapter VII.) between the hot spent liquor and softened water intended for boiler feed purposes. Apart from recovering a sensible quantity of heat in this way, it is possible to recover a large portion of the ammonia content of the effluent liquor.

Fig. 151 gives details of an apparatus which functions in the dual capacity. This arrangement of plant is the subject of patent No. E.P. 177726. It comprises a tubular feed-water heater *a*, having above its upper tube plate a perforated distributing plate *a*₁, on to which is discharged through pipe *b* the hot waste effluent from the fixed ammonia still, the effluent being kept at this level by a rising out-flow pipe *c*. Surmounting this heater is a chamber *d*, preferably suitably lagged as at *e*, and having as a part of one side of it the tube plate *f* of a multitubular condenser which discharges into a closed receiver *g*.

The ammoniacal steam rising from the surface of the liquor in the chamber condenses in the condenser and thus a reduced pressure is maintained within the chamber; when a certain amount of condensate has collected in the receiver *g*, as indicated by a gauge glass *h*, valve *i* in pipe *k* is opened to allow the condensate to flow into a tank.

l is a trap which prevents return of condensate to the chamber when the receiver is more than normally charged. The feed water enters the heater *a* at *m* and leaves it at *n*.

Where heat interchange is not decided upon, several methods of cooling are

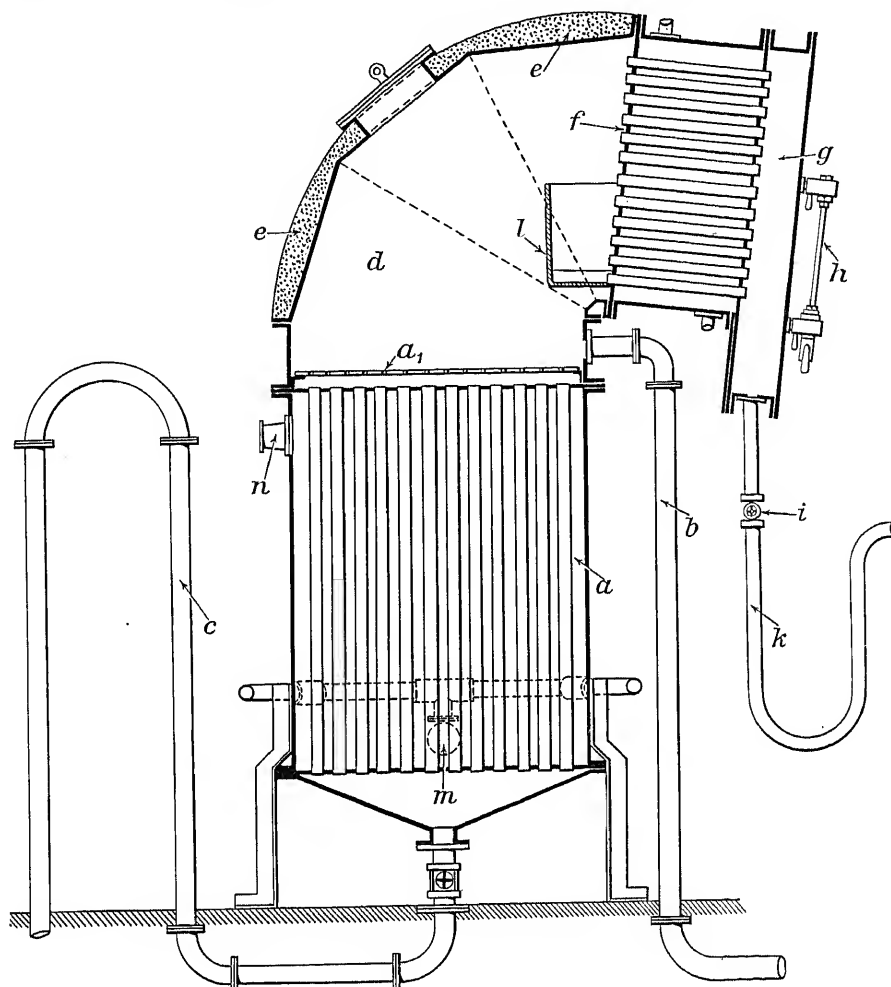


FIG. 151.—FEED WATER HEATER.

available, and the choice of a method is generally determined by the size of the plant and local circumstances.

COOLING ARRANGEMENTS FOR VARIOUS SIZES OF PLANT

In connection with small works it is usual to build a concrete tank with a series of baffles, and to pass the spent liquor from the still through two or three series of wrought-iron flanged pipes, having suitable provision for cleaning, which are immersed in water circulated through the baffles in counter-current flow to the passage of the hot spent liquor.

At some small and medium-sized works a cast-iron serpentine cooler is used. This

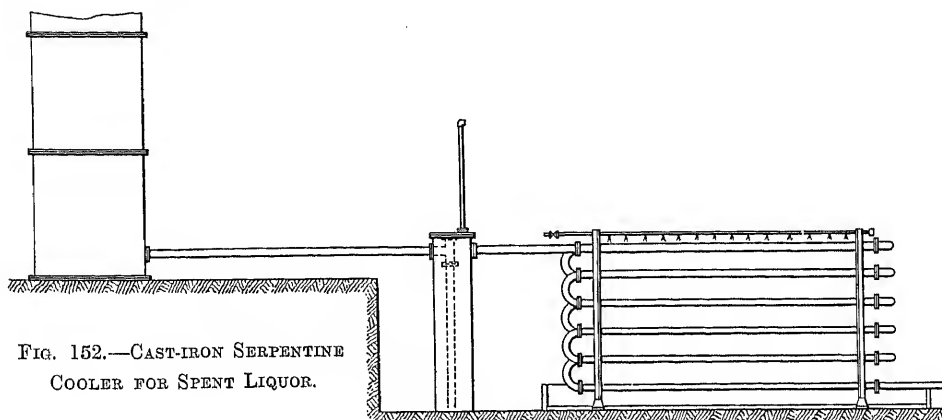


FIG. 152.—CAST-IRON SERPENTINE COOLER FOR SPENT LIQUOR.

is carried by vertical angle iron supports in a concrete pit. The serpentine is cooled by an efficient type of water spray. Fig. 152 illustrates this type of arrangement.

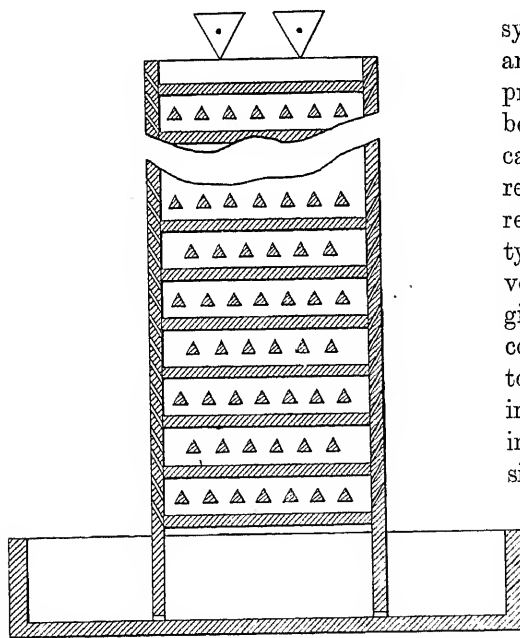


FIG. 153.—VENETIAN TYPE OF SPLASH COOLER.

At some large works the decanter system of sludge removal is in vogue, and in these cases it is the invariable practice to remove the sludge first before cooling. Details of these decanters and their operation will be referred to later. Where the sludge is removed initially, the Venetian blind type of splash cooler is adopted with very satisfactory results. Fig. 153 gives the details of this type of cooling tower. Two of the sides of the tower are provided with boards, fixed in the form of Venetian blinds. The internal elements of the tower consist simply of triangular bars or inverted V's, staggered in such a way as to permit of a fine division of the water so as to aid cooling. A tumbler device is used for distributing the liquor, which is collected in a lead-lined dish at the foot, in which the cooling

tower stands. It has been found necessary at some works to prevent an undue discharge of steam during the cooling operations referred to. In such cases draughting devices must be adopted.

METHODS OF COLLECTING THE SLUDGE

In general, three methods are in operation for the collection of the lime sludge discharged with the spent liquor from ammonia stills: (1) settlement in pits, (2) settlement in decanters, and (3) filter pressing.

1. *Settlement in Pits*

This is the general method in use at most works, whether small, medium-sized,

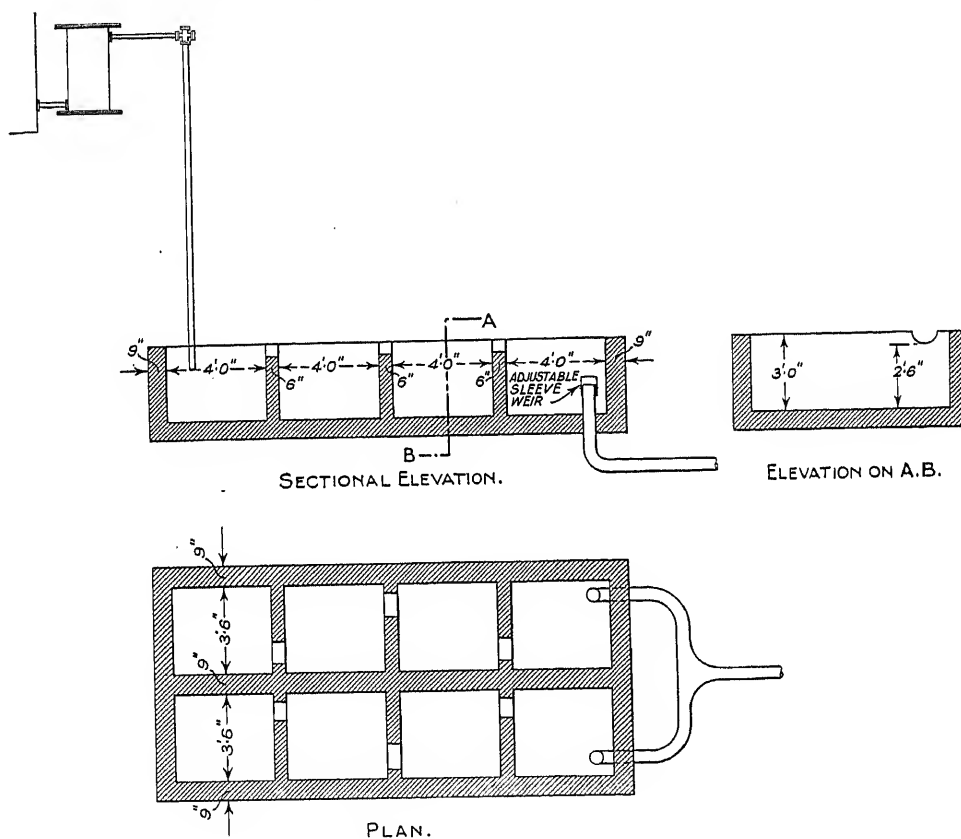


FIG. 154.—SETTLING PITS FOR LIME SLUDGE.

or large. At small works it is usual to build two or more concrete pits, equipped with baffle walls, and to work these alternately, accumulating sludge in one and emptying the drained deposit from the other. The extent of the travel afforded by the baffles is such that perfectly clear spent liquor is discharged and the lime sludge retained. It is usual to provide an adjustable weir, or other device, at the outlet to the sewer. Fig. 154 gives details and dimensions of a sludge settlement arrangement suitable for a works distilling 3500 to 5000 gallons of gas liquor per day.

Figs. 155 and 156 show the provision made for settling sludge from spent liquor arising from the distillation of 200,000 gallons of gas liquor per day, and the method adopted for the removal of the deposited sludge. The light railway track can be

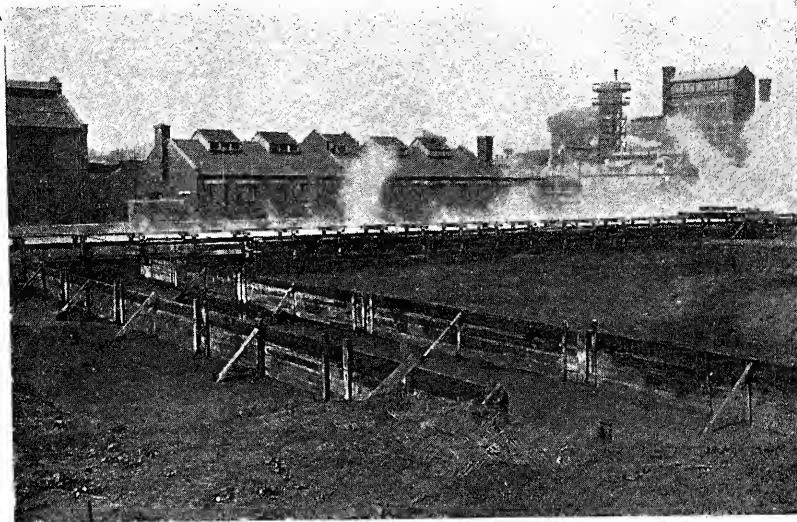


FIG. 155.—LARGE PLANT FOR SETTLEMENT OF LIME SLUDGE.

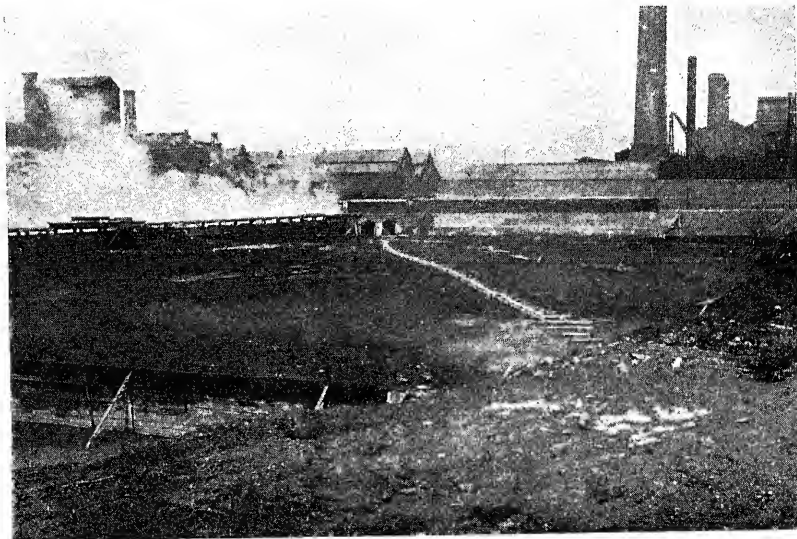


FIG. 156.—LARGE PLANT FOR SETTLEMENT OF LIME SLUDGE.

readily removed to any part of the pit, and can be extended at will. This method of operation is convenient, and does not entail more than one shilling per ton, which includes the loading of the sludge to barges.

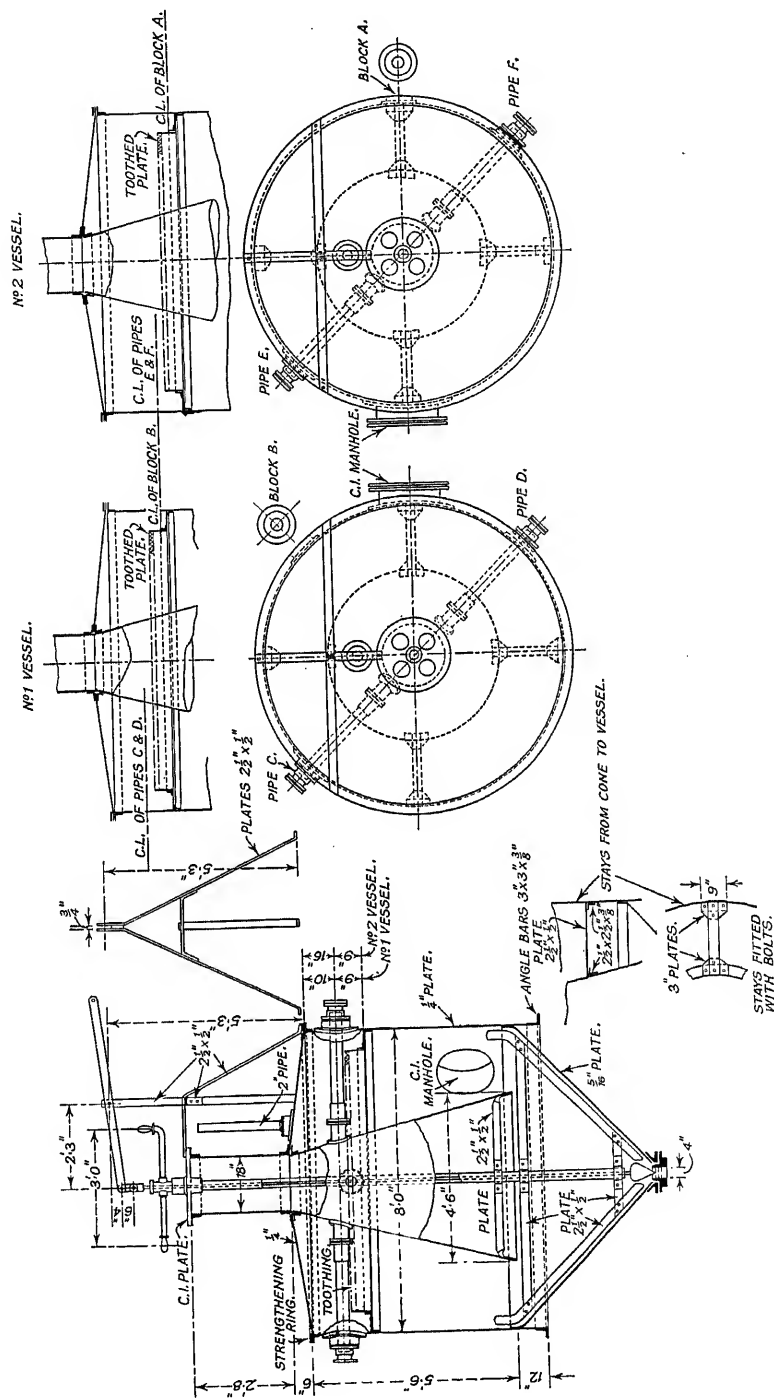


Fig. 157.—LIME DECANTERS.

2. Settlement in Decanters

Some works prefer to adopt decanters of the form shown in fig. 157. The still in conjunction with which the decanters work is usually fixed on a base high enough to allow the decanters to be filled by gravity without imposing any extra pressure on the still, as would possibly result if the decanters were placed on the same level as the still. It will be seen that the decanters consist of conical steel vessels, to which four legs are attached. They are generally placed in a concrete pit. The hot spent liquor is run direct from the still through a seal pipe, and enters the inner cone at the side near the top. The liquor descends, passing under the base of the inner

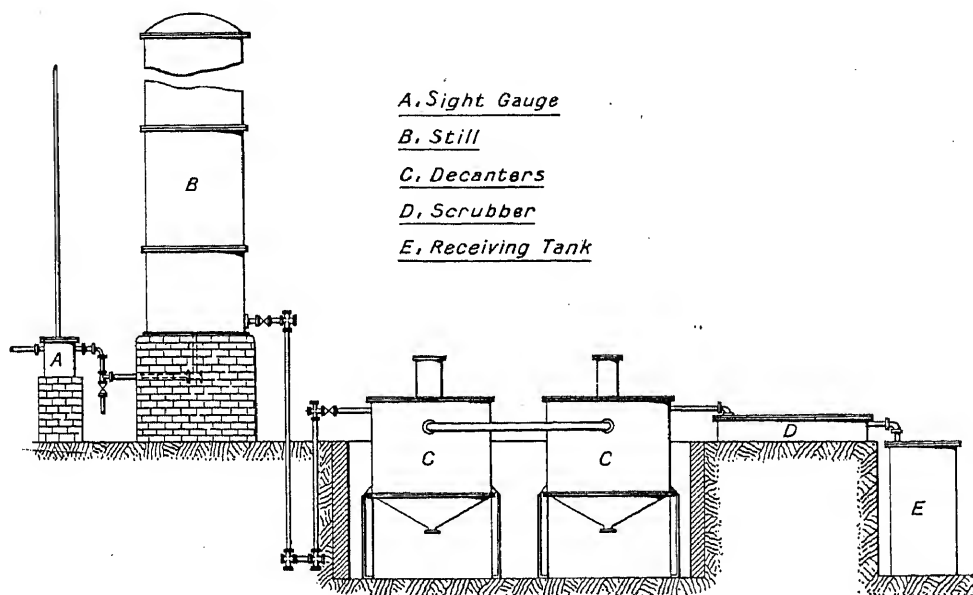


FIG. 158.—POSITION OF DECANTERS IN RELATION TO STILL.

cone, and gradually rises, filling completely the vessel until it overflows *via* the serrated notches to the outlet pipe which is near the top of the outer shell.

Each of the decanters is provided with a vent, and the steam and ammonia evolved pass to a scrubber fed with water, the resulting liquor being sent to the gas liquor storage tanks.

Four such decanters are used in connection with a plant distilling approximately 25,000 gallons of liquor per day. Two are constantly receiving the spent liquor, and the other two are either being discharged of the sludge or are ready preparatory to the receipt of further quantities of effluent liquor. It is usual to discharge the decanters once each day. Prior to their discharge, the scraping device is operated, so as to remove any adhering sludge from the sides of the vessel. The sludge is dumped on hot boiler ashes, thus causing a further evaporation of water and ensuring that the mixture is in a reasonably dry condition for manipulation.

Fig. 158 shows the position of the decanters in relation to the still. After leaving the decanters the effluent liquor is passed through an open filter box, packed with graded ashes, to arrest any slight traces of sludge which may be entrained, and subsequently is cooled preparatory to being finally discharged.

3. *Filter Pressing of the Sludge*

With the two methods of settlement referred to, the content of moisture in the sludge cannot be reduced much below 60 per cent. This constitutes a distinct disadvantage, especially when the sludge has to be carted or removed to a dump. By adopting filter presses it is possible to reduce the moisture content to about 30 per cent. This represents an appreciable saving in transit charges. There are, however, many considerations to be borne in mind. Usually the labour expenses are higher with this method, besides which there is expenditure on filter cloths, pumping, and general wear and tear of additional plant.

Apart from the adoption of concrete baffle pits it is necessary to adopt two other concrete tanks. The need for these will be seen later. The settled sludge, associated with which is about 60 per cent of water, when taken from the settling pits, should not be sent direct to the filter presses, but should be discharged by suitable means (pneumatic forcing rams or otherwise), into the additional settling tanks referred to, where the sludge must be diluted with three times its volume of cold water. After a further settlement in one or other of these tanks the sludge can be filter-pressed in the usual manner.

The necessity for additional dilution with water and subsequent settling is to cool the sludge, and to reduce its alkalinity to a minimum. If these precautions are not observed it will be found that considerable expense will be entailed by reason of the renewal of filter cloths. Indeed, the expenditure will be such as to nullify any advantage which may otherwise be derived from this method of treatment.

Automatic sludge-forcing rams, which work pneumatically, are recommended by reason of their economical operation. The air is initially exhausted from one ram, so that the sludge is drawn in until the ram is filled. The air valves are then reversed so that the compressed air is pumped into the latter, thus forcing the sludge out through the delivery valves wherever the sludge is required. While it is being forced from one ram, the other ram is being filled by vacuum. Thus the rams work alternately, one filling while the other is discharging, thereby maintaining a constant flow. The apparatus works with a constant amount of air, which, by means of a combined air compressor and vacuum pump, is forced at first into one ram and then into the other as required. The reversal of the air pressure at each charge is automatic, so that the apparatus requires very little attention.

The air compressor need not be adjacent to the rams, but may be situated in any convenient place (say) in the power plant house.

The automatic sludge-forcing rams are also used for filling the filter presses, and are well adapted for delivering a semi-liquid under pressure.

TREATMENT OF THE EFFLUENT SPENT LIQUOR

The spent liquor, after the elimination of the sludge, is noxious in character, when judged by ordinary standards of purity.

Before proceeding to the methods of treatment of the effluent spent liquor, it is advisable to consider how far such liquor is noxious when contrasted with the standard of purity recognised by the river and sewage authorities.

From the Eighth Report of the Royal Commission on Sewage Disposal, it is possible to arrive at what is ordinarily accepted as a standard of purity. The report in question says:

"If one hundred thousand cubic centimetres of river water do not normally take up more than 0.4 grain of dissolved oxygen in five days, the river will ordinarily be free from signs of pollution, and if a river water usually gives a higher figure than this it will almost certainly show signs of pollution, except perhaps in very cold weather. This figure (0.4) we term the 'limiting figure,' and in our opinion it should be the foundation on which any scheme of standards should be constructed.

"Of the various conditions which should be taken into account in considering the question of standards, by far the most important in our view is the *degree of dilution* afforded by a river to a contaminating discharge. The importance of this factor is sufficiently shown by the numerous instances in which the degree of dilution is sufficient to enable practically the whole of the work of sewage purification to be done by natural agencies without cost or injury to the community—assuming that the diluting water is of reasonable purity, we shall expect to find in the degree of dilution a measure alike of the amount of purification which may be safely entrusted to the river, and of the amount of purification of the polluting liquid which could be effected prior to the liquid being discharged."

TOXICITY OF EFFLUENT LIQUOR AND THE FACTORS GOVERNING IT

It will be appreciated that the toxicity of effluent liquors varies sensibly, and depends very largely on the time during which tar has been in contact with the liquor, and on the efficiency of the separation of the tar from the liquor at the gas works or coke ovens. Moreover, different systems of carbonisation may conceivably exercise a marked influence on the character of the gas liquor, and hence the resulting effluent liquor. There is reason to believe that at those works where vertical retorts are used for the carbonisation of coal, owing to the quality of the resultant tar, and the arrangements for separation, the effluent liquor arising from the distillation of the gas liquor produced at such works is more toxic than is the liquor emanating from gas works carbonising coal in horizontal retorts.

In confirmation of the foregoing view the following figures are interesting:

Basis of Analyses—Three-minutes Oxygen Test

- | | |
|---------------------------------------|--|
| 1. Effluent liquor resulting from gas | 560 parts of O ₂ absorbed per 100,000 |
| liquor arising from gas works | gallons. |
| using vertical retorts. | |

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- | | |
|---|---|
| 2. Effluent liquor resulting from gas liquor arising from gas works using horizontal retorts. | Varying between 200 and 350 parts of O ₂ absorbed per 100,000 gallons. |
| 3. Devil liquor arising from distillation of No. 2 gas liquor. | Varying between 375 and 410 parts of O ₂ absorbed per 100,000 gallons. |

A valuable contribution on the presence of polyhydric phenols in ammoniacal liquor derived from carbonisation of coal has been published by G. S. Currey (*Chemistry and Industry*, 1923, 379T.). The investigation was carried out with a view to elucidating a red brown coloration given by the effluent to the Paramatta River, near the Australian Mortlake Gas Works. The coloration in question was found to be due to the oxidation of polyhydric phenols, chiefly catachol.

The Oldbury District Council suffered very acutely about 1905 from effluent spent liquors received at the sewage works, and entrusted the study of ascertaining the "best practicable means of meeting the difficulties they encountered" to Prof. Percy F. Frankland, F.R.S., who in February 1907 contributed the results of the protracted inquiries by himself and H. Silvester to the Birmingham and Midland Section of the Society of Chemical Industry (*Journal*, 1907, pp. 231-237). This contribution added materially to the knowledge of that time on a hitherto obscure subject, and it is important, even to-day, to direct attention to a few of the outstanding conclusions reached.

Prior to the investigations in question, it was generally believed that sulphocyanide was the most toxic constituent of spent liquor, but from experiments made by Frankland it was shown that goldfish could live for a period of at least twenty-four hours without apparent discomfort, in solutions containing up to 250 parts of sulphocyanide (CNS) per hundred thousand. Signs of distress were evident at 450 parts, and at 500 parts the goldfish died.

An average analysis of five samples of spent gas liquor revealed the amount of sulphocyanide to be only 153.8 parts per hundred thousand, the maximum being 171.7 parts. If sulphocyanide had been the only constituent in effluent liquor noxious to goldfish life, no objection need be urged, as the average content in the sewage containing such liquor was only 9.3 parts. It was found, however, that sulphocyanide prejudicially affected *Bacillus coli communis* in amounts as low as 10 parts per hundred thousand. But as the investigations were pursued, it was soon discovered that there were other oxygen-consuming bodies of far greater moment than sulphocyanide, though this body had been regarded hitherto as a veritable curse.

It was found that phenols constituted a much more toxic agent than sulphocyanide, and the amount so formed was nearly equal to the latter. It was also discovered that sulphocyanide is not destroyed in the septic tank, and that the "oxygen absorbed" is apparently not reduced there either.

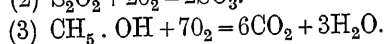
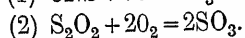
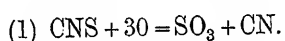
To determine the amount of oxygen required for the oxidation of the chief constituents of spent gas liquors, the estimation was conducted in precisely the same way as is customary in ascertaining the oxygen absorbed in ordinary sewage,

namely, four hours' test at 80° F. The following figures were furnished in this connection :

TABLE 56

No.	100 Parts of Ingredients.	Amount of Oxygen required (Theoretical).	Oxygen absorbed in 4 Hours at 80° F. (Experimental).	Percentage of Theoretical obtained in Experiment.
1	Sulphocyanide (CNS) .	82.7	82.7	100.0
2	Thiosulphate (S ₂) . .	100.0	85.6	85.6
3	Phenol (C ₆ H ₅ . OH) .	238.0	178.0	74.8

The undermentioned reactions were assumed :



Experiments indicated that sulphocyanide apparently undergoes no change in an effluent containing an adequacy of nitric nitrogen, even in the course of four days at about 20° C.

The work to be effected by the Oldbury bacteria beds is shown by the authors in the following tables :

TABLE 57

Ingredients in Twenty-four Hours' Flow (Maximum).	Weight.	Weight of Oxygen required for Complete Oxidation.	Volume of Oxygen required.
	cwt.	cwt.	cub. ft.
Sulphocyanide (CNS) .	9.60	8.0	10,000
Phenol (C ₆ H ₅ . OH) .	8.75	20.8	26,065
Thiosulphate (S ₂) . .	2.90	2.9	3,665
	21.25	31.7	39,730

A remarkable factor bearing upon the foregoing experiments was elucidated by F. W. Skirrow, and published in his paper on "The Determination of Phenols in Gas Liquor" (*Journal Society of Chemical Industry*, 1908, pp. 58-61). It was shown by Skirrow that the amount of pure phenol which is lost in ammonia liquors amounts

to between 0.37 and 0.55 lb. per ton of coal carbonised, whereas the amount of pure phenol recoverable from the tar is only 0.53 lb. per ton. In other words, about one half of the pure phenol which is produced in the retorts is lost in the ammoniacal liquor.

BACTERIAL TREATMENT OF EFFLUENT SPENT LIQUOR

Dr. Fowler was responsible for the pioneer work in the bacterial treatment of effluent liquor. On this subject, the 44th Report of the Chief Alkali Inspector says: "For this purpose a certain number of bacteria from the Manchester Corporation sewage works were indentured, separated from their families, and transported to the Bradford Road residual works of the Corporation, and there immured in a tower filter, of simple construction, as their working home, not a 'compound.' The task assigned to them, however, was the breaking-up of 'compounds.'" These experiments were conducted for several years; the filter started in June 1904. It will be realised that many data had to be ascertained, such as the lowest permissible amount of dilution of the spent liquor, compatible with the maintenance of the bacteria in an efficient and thriving condition for accomplishing their work in the minimum of time. By conducting experiments the degree of concentration was reached at which the bacteria sickened and were incapable of continuing their work till former conditions were restored by a period of rest, followed by a diminution in the strength of the applied liquor. It was subsequently found that the effluent, after treatment, could itself be applied as a diluent in lieu of fresh water.

THE FOWLER SYSTEM—LARGE-SCALE OPERATION

The Fowler system of purification was put into operation at the Bradford Corporation Chemical Works, Frizinghall, about 1908, and has been in operation since. The following account of the Frizinghall filter beds and their method of working has been prepared from details furnished at different times by Percy Ward, the engineer and manager of the chemical works in question. The filters cover half an acre in extent, and are constructed of graded clinker arising from the steam boiler plant. The bottom layer, to the extent of 1 ft. deep, is of 4 to 6 in. in size, above which is a layer of about 1 ft. from 1 to 2 in. in size, and finally, there is a layer of 6 ft. from half to 1 in. in size.

Only one bacterial bed is provided at Frizinghall. It is possible to construct bacterial beds without the erection of retaining walls, merely allowing the ashes to seek their natural angle of repose. Supernatant effluent liquor divested of lime sludge, diluted by fifteen times its own volume of water, is pumped over the bed, which initially has been treated with stable or human urine, sprayed on the top of the bed day by day for a fortnight.

As soon as the effluent liquor leaving the bacterial beds shows a purification of 80 per cent, this liquor is returned for the dilution of the untreated spent liquor, thus dispensing with the use of water, which may or may not be expensive, according to circumstances. The effluent liquor needs to be uniformly distributed over the

bacterial bed by a suitable self-propelled device such as is ordinarily used at sewage works. As the bacteria sicken, an appropriate rest period is necessary, and this is secured at the week-ends, etc.

In the course of time, also, a deposit of lime salts forms on the interior of the connecting pipes of the filter, and pending cleaning, the circulation is sensibly reduced, involving a much higher circulating liquor. There is no appreciable reduction in the efficiency of purification during this period.

The oxygen test of the original spent liquor varies around 560 parts per hundred thousand; the purified liquor is 110 parts—a difference of 450 parts. The feed to the filters is about 140 parts, and the liquor leaving the filters is 110 parts. It will

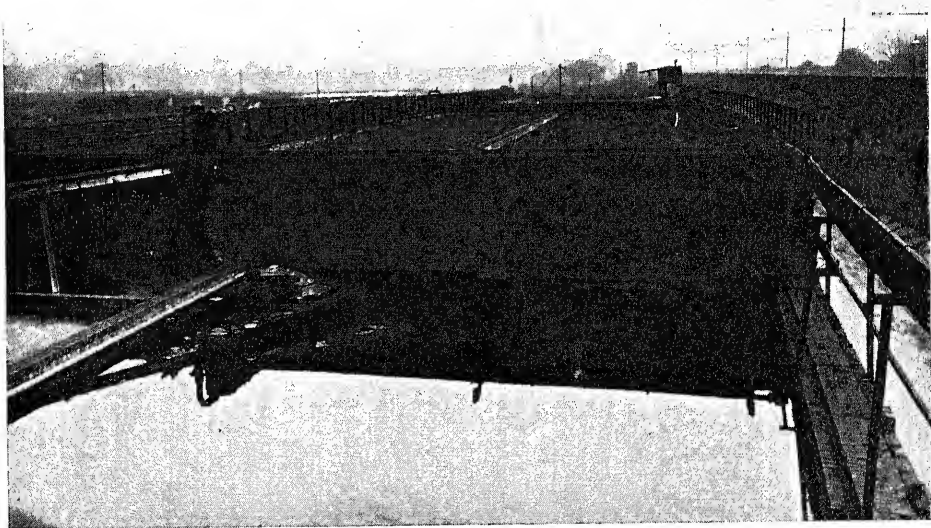


FIG. 159.—BACTERIAL BED FOR THE PURIFICATION OF SLUDGE-FREED EFFLUENT LIQUOR.

be appreciated from what has been said that to determine the amount of purification in the case cited the difference between the last two tests requires multiplying by 15.

Each year it is found necessary to turn over with a fork the top eight inches of filter material, owing to calcium deposits having a caking effect to this depth.

The importance of good distribution cannot be too strongly urged as a factor in securing efficient purification. It should be stated that at Frizinghall, approximately thirty thousand gallons of effluent liquor are treated per day. Fig. 159 gives a view of the bacterial bed just described.

As an indication of the size of the filter beds for the Fowler bacterial treatment of effluent liquor, it may be said that there should preferably be two beds, each of which should be 35 ft. by 35 ft. per ton of sulphate of ammonia manufactured per

day. Owing to the isolated position of the Frizinghall plant the machinery consists of a gas engine and a centrifugal pump in duplicate. With gas at four shillings per thousand cubic feet, the cost of pumping and circulating the effluent liquor as described represents approximately two shillings per ton of sulphate of ammonia manufactured.

The plant is closed down at week-ends and about one month each year, during which time gas liquor is accumulated at the several gas works. It is emphasised that this rest period is very beneficial to the bacterial life in the filter.

It will perhaps be interesting to give the tests applied for determining the oxygen absorption of effluent spent liquor.

THREE-MINUTE OXYGEN TEST

Solutions required :

N/80 KMnO_4 . (1 c.c. = 0.0001 gr. O).	10 per cent H_2SO_4 .
N/80 $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$.	Starch solution.

20 c.c. water are taken in small wide-necked flask and acidulated with 10 per cent H_2SO_4 . The KMnO_4 is then run in drop by drop until a faint but permanent colour is obtained. Then in the case of (a) raw waste liquor, a further 50/60 c.c. of permanganate solution are run in ; in the case of (b) treated liquor, about 20 c.c. When testing (a) use 1 c.c. of liquor, and (b) 1 to 5 c.c., as a trial test will show. After the addition, shake occasionally during the three minutes. Then quickly add a small crystal of potassium iodide and titrate back with the thiosulphate solution until the yellow colour nearly disappears, add starch solution and complete (*i.e.* until blue colour disappears).

From the permanganate and thiosulphate used one can find the permanganate used by the liquor, and thus the oxygen consumed, which is calculated to parts per 100,000.

EXAMPLES ILLUSTRATIVE OF THE CALCULATIONS INVOLVED IN THE TESTS

1 c.c. Raw waste liquor taken.

60 c.c. N/80 KMnO_4 .

20 c.c. N/80 $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ used in backward titration.

Difference 40 c.c. N/80 KMnO_4 consumed,

= 40×0.0001 grm. oxygen,

= 0.0040 grm. oxygen.

= 400 parts per 100,000.

5 c.c. treated waste liquor.

20 c.c. N/80 KMnO_4 .

1.4 c.c. N/80 $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ used in backward titration.

Difference 18.6 c.c. N/80 KMnO_4 consumed.

= 18.6×0.0001 gr. oxygen.

= 0.00186 gr. oxygen,

= 186 parts per 500,000.

= 37.2 parts per 100,000.

DECOLORISING AND DEPHENOLATING METHOD

In the last two Annual Reports (1921 and 1922) of the Chief Alkali Inspector, reference has been made by Dr. T. Lewis Bailey to a method by which phenols (the most noxious constituents of effluent spent liquors, as has already been remarked) are removable from solutions by steam distillation. The phenoloid constituents can be largely eliminated by making hot fire gases ascend by means of a steam injector, a suitable scrubber, down which the spent liquor is distributed.

No practicable method of removing thiocyanate has yet been found, and the elimination of thiosulphate is no less difficult. Both these bodies are comparatively stable, and oxidation by air in dilute solutions is next to impossible.

It has already been pointed out in Chapter III. that thiocyanate and thiosulphate are generally looked upon as oxidation products, and the content of these constituents in ammoniacal liquor is a function of the contact of the liquor with oxygen in the condensing and scrubbing systems of the coal gas plant.

It has been indicated by Dr. Bailey that a correct knowledge of the quantity of spent liquor produced in relation to the liquor distilled is fundamental to a consideration of the treatment of effluent by blowing with fire gases.

Figures based on determinations by the author are given by the Chief Alkali Inspector in the 1922 report, and these figures are reproduced below :

TABLE 58
VOLUME OF EFFLUENT SPENT LIQUOR IN RELATION TO VOLUME
OF FEED LIQUOR

Strength of Gas Liquor.		Gallons of Liquor per Ton of Ammonium Sulphate, based on 100 per cent Conversion. NH_3 content of S/A - 25 per cent.	
Oz. Strength.	Per Cent NH_3 .	Gas Liquor used.	Total Effluent Spent Liquor calculated for 50 per cent increase on G/L.
4	0.868	6470	9710
5	1.085	5170	7760
6	1.302	4320	6480
7	1.519	3690	5540
8	1.730	3230	4850
9	1.953	2870	4310
10	2.170	2580	3870
11	2.389	2340	3510
12	2.604	2150	3230

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It is remarked that the above figures are consistent with those given by Dr. Maclean Wilson for average coke oven practice. The figures applying to coke ovens are appended.

TABLE 59

20,000 gallons of effluent spent liquor are stated to be discharged from works carbonising 200 tons of dry coal per day, corresponding to about 9000 gallons per ton of S/A made. Of this :

	Average.	Per 100 Vols. of Total Effluent.
Water of combustion accounts for	9	} = 65 (Liquor distilled).
Moisture in the coal accounts for	23	
Water used on the scrubbers accounts for	33	
Water condensed from steam used.	17	} = 35 (Added in still).
Lime water feed	18	
	<hr/> 100 <hr/>	

Whence :

Vol. of final effluent : Vol. of feed : : 100 : 65 (= 154 : 100).

It is usually assumed, judging by the general character of the liquors, that the proportion of phenoloid bodies will be relatively higher in coke oven liquors than in those produced at gas works. An important consideration bearing upon the phenol content of effluent liquor is that of the devil liquor resulting in the distillation. According to tests made by the author the devil liquor is more often than not 50 per cent more toxic than the effluent liquor, and at times even 100 per cent more toxic. It is therefore a matter for consideration whether it would not be desirable to submit the devil liquor to separate treatment by evaporation, say, by exhaust steam,—a practice which is followed by some gas works within the author's knowledge.

In this connection it is important to bear in mind that it is possible to separate from the devil liquor an oleaginous body which is known in works parlance as "devil oil." Quantities of this oil are also collected from the seal pots of the waste gas mains prior to the gases being introduced to the oxide of iron heaps, Claus kiln, or spent oxide burners, according to the practice followed. An analysis of devil oil made by W. Kirby, B.Sc., is subjoined below, together with some observations which may be of interest :—

ANALYSIS OF DEVIL OIL

Sp. gr. 1.015

Pyridine bases	42	per cent.
Tar Acids	13	,, ,,
Benzonitrile	5	,, ,,
Neutral Oils	31	,, ,,
Naphthalene	9	,, ,,

The pyridine bases consist of approximately 40 per cent pyridine, the remaining 60 per cent being constituted of picolines and lutidines. Contrary to expectations the tar acids contain very little phenol and consist almost wholly of cresols.

The neutral oils contain traces of toluene and xylenes; the boiling range is similar to that of a creosote fraction from coal tar. Devil oil is saturated with a number of poisonous gases—sulphuretted hydrogen, hydrogen cyanide and carbon dioxide, which help to give it a penetrating sickly odour. A peculiarity of this odour is its extreme persistence where actual contact with the oil has taken place.

The colour of spent liquor on discharge from the stills is pale straw, but on exposure to the air it becomes dark brown. The coloration has relation to the presence of oxidisable organic bodies, and only reveals itself in alkaline solution. Dr. Bailey remarks that the effect of passing carbon dioxide through the effluent liquor has been tried in several cases with gas works liquor, but in no case was the colour discharged, neither was there any discernible separation of lime salts in either hot or cold solution. It is known that a sensible proportion of the alkalinity of effluent liquor is due to traces of unvolatilised ammonia and pyridine. Boiling will remove the latter constituent, but the remaining alkalinity is fixed, due to the presence of free lime. Summarising the results of a series of experiments, Dr. Bailey says:

“(1) Volatilisation of phenol is poor with a liquor feed of 7.6 c.c. per minute (Expt. No. 1 (a)). The relatively small amounts of liquor vaporised in B and condensed in C indicate that contact of liquor and steam was imperfect—cold liquor appears to have descended on one side and steam and air ascended on the other.

“(2) Volatilisation of phenol is satisfactory in Expts. 1 (b) and 2 (a), but the high exit temperature of B (96-97° C.) has led to undue volatilisation of water.

“(3) The effect of doubling the rate of air current in promoting volatilisation of phenol is seen in Expt. 2 (c), where the efficiency is 91 per cent with a scrubber exit temperature of 84° C., as against 68 per cent with an exit temperature of 91° C. in Expt. 2 (b).

“(4) Comparison of the relative weights of phenol condensed in C and D respectively indicates that phenol has a perceptible vapour tension in aqueous solution at 80-85° C., even at the low concentration of 0.1 gram. C_6H_5OH per 100 c.c. (Expt. 2 (a)).

“The size of the scrubber needed to deal with four thousand gallons of effluent per twenty-four hours, which is the amount produced in the manufacture of one ton of sulphate of ammonia, is given, basing on a 90 per cent efficiency, as follows:

“Phenol in liquor, 0.119 lb. per 10 gallons = 47.6 lb. per 24 hours.

90 per cent of this gives the amount to be volatilised = 42.8 lb.

13 litres or 0.46 cubic feet of air will volatilise 0.098 gram. = 0.000216 lb. phenol.

Whence, volume of boiler fire gases (0° C. and 760 mm.) required to vaporise 42.8 lb. phenol } = 91,000 cubic feet per 24 hours, or 1.05 cubic feet per second.

"Of this volume, 28,000 cub. ft., or less than one-third, would suffice to decolorise the effluent.

"As regards the size of scrubber needed to effect the treatment, it may be calculated from the apparatus used that a scrubber 16 ft. high by 3 ft. diameter, packed for 13 ft., would serve quite well.

"Confirmatory data were obtained at the works by means of an apparatus of laboratory dimensions erected at the boiler flue by the works chemist, and operated by him under conditions similar to those observed in the experiments described above.

"The apparatus consisted of a metal cylinder 18 in. high by 5 in. diameter (=0.205 cub. ft.), packed with small pieces of hard coke. Effluent spent liquor from the settling pits was fed on to the scrubber hot (80° C.), and in its descent it met an upward current of boiler fire gases (total acidity equivalent to about 1 grain H_2SO_4 per cub. ft.), driven in at the base of the scrubber by a Bunsen pump operating as a steam injector. The volatilised phenol and water vapour escaped into the air from the top of the scrubber.

"Working thus at different rates of flow, it was found that the maximum rate of feed consistent with due decolorisation of the liquor was 160 c.c. per minute, or 240 gallons of liquor per cub. ft. of packing per 24 hours."

DEPHENOLATING PROCESS—LARGE-SCALE OPERATION

Large-scale experiments, based on the above preliminary trials, were put into operation at the Hornsey Gas Works by J. W. Buckley, the engineer. In course of time this plant developed into two scrubbers, apparently old boiler tubes, with accessory parts, steam injector, and connections. Both scrubbers were lagged in order to aid more efficient volatilisation of the phenoloid bodies.

The use of a fan at the outlet of the scrubbers has been recommended. This fan would be used to pull flue gases through the latter apparatus under suction.

Given an adequate volume of flue gases, there is reason to believe that the major portion of the phenols can be removed by volatilisation. Indeed, with the plant at Hornsey, treating the whole of the effluent liquors produced in the manufacture of $2\frac{1}{2}$ -3 tons of sulphate of ammonia per twenty-four hours, it seems from results indicated by Dr. Bailey in the 1922 Alkali Report that 71 per cent of the phenols were vaporised, with a reduction in the "oxygen absorbed" figure of 42 per cent.

Careful consideration suggests that the merit of the dephenolating process resides in the removal of the major portion of the phenols, which may account for from 50 to 70 per cent of the "oxygen absorbed." Such a process, working in conjunction with bacterial beds, which would then be largely confined to the treatment of the thiocyanate and thiosulphate contents of the effluent liquor, would undoubtedly constitute a very satisfactory process, calculated to give a tolerably innocuous spent liquor. Of course, it will be realised that the economics of the combined processes will need to be considered.

LIME SLUDGE

Analyses of the lime sludge produced at various works will vary widely. If lime is being used efficiently, the major portion of the sludge should be calcium sulphate, with a little calcium carbonate, due to atmospheric carbonation, and a little free lime as hydrate. At one ammonium sulphate works, the sludge arising from which the author had occasion to examine, it was found that an analysis of the dried material gave the following results :

TABLE 60

CaSO ₄	.	.	.	24.00	per cent.
CaCO ₃	.	.	.	34.77	„ „
CaO	.	.	.	39.35	„ „
SiO ₂	.	.	.	1.42	„ „
				99.54	„ „

The calcium carbonate and calcium oxide figures are far too high, and indicate that an excess of lime was being used. Attention has already been directed to the importance of carefully controlling the feed of lime to the stills in Chapter VIII., but it is interesting to consider what a slight excess of free lime in the effluent liquor means. Consider a works making 3 tons of sulphate of ammonia 25.5 per cent NH₃ per day from gas liquor having a strength of 8 oz. (1.73 per cent NH₃), and basing on a 95 per cent efficiency, 11,416 gallons will need to be distilled, and the liquor emerging from the stills as effluent spent liquor will be about 50 per cent in addition, namely 17,124 gallons, representing a total weight of 76.4 tons.

Now 0.1 per cent of free lime in such an effluent will represent 1.528 cwt. As the theoretical lime consumption, based on 20 per cent fixed ammonia, is 0.255 ton NH₃

$$\times 3 \times \frac{100}{95} \times \frac{1}{5} = 3.22 \text{ cwt. fixed ammonia, or } 5.3 \text{ cwt. CaO, it will be seen that a small}$$

excess of 0.1 per cent of free lime in the effluent liquor represents a surplus of lime of 28 per cent. If the formation of unnecessary sludge is to be avoided, attention to the feed of lime to the stills cannot be too vigilant. One ton of excess lime represents 2½ tons of sludge, when based on a 60 per cent content of water (which is a usual content in lime sludge).

Occasional analyses of the lime deposit are desirable, as affording an extra check on the free calcium hydrate content of the effluent liquor.

The question of the use of lime sludge on the land has often been raised, but as far as can be ascertained no definite or sustained trials have been made. Its utility depends on the easy oxidisability of the associated compounds. It is not inconceivable that this material may be used for partial sterilisation, as it contains traces of naphthalene and phenoloid bodies. Of course, to be effective for this purpose, the protozoa in the soil and a good many of the bacteria must be destroyed, but the

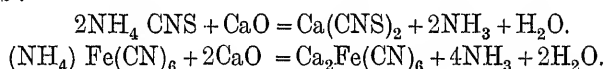
spores of these would probably be retained in a living state, and would ultimately become active. This would mean that the injurious effects of the bodies named in a waste product such as lime sludge must have disappeared before any cultivated crop was put on the land.

The best use of lime sludge would appear to lie in the production of mortar, or as a raw product in the manufacture of cement, or bricks.

THE RECOVERY OF FERROCYANIDES AND SULPHOCYANIDES FROM EFFLUENT LIQUOR

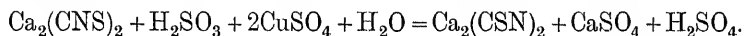
As has been remarked, many processes have been suggested for the recovery of ferrocyanides and sulphocyanides from the effluent spent liquor, but none appears to have gained great popularity.

The ferrocyanides and sulphocyanides present in gas liquor exist as fixed ammonium compounds, and are decomposed in the fixed ammonia still by the use of milk of lime. Calcium ferrocyanide and sulphocyanide are formed, and ammonia is liberated thus :



In one method of recovery the effluent liquor was run into a tank, from which it was pumped to the top of a tower filled with coke or other suitable filling. Irrigation of the packing medium was effected, and sulphur dioxide and carbonic acid were passed through the tower. In this way the alkalinity of the liquor was destroyed, and a slight acidity secured. The liquor leaving the base of the scrubber passed through a weir into one of the larger compartments of a settling tank. Here the effluent was treated with ferrous and ferric salts, and in this way the ferrocyanide was precipitated as Prussian blue.

After settlement, the supernatant liquor was run off and treated for the recovery of sulphocyanides. The only suitable insoluble sulphocyanide, from a practical point of view, is that of copper. Hence copper was the medium by which the recovery was effected. In a second tank a solution of a salt of copper—copper sulphate—was added, and the insoluble white cupreous sulphocyanide precipitated thus :



Assuming the liquor had not been treated initially with sulphurous acid gas, black cupric sulphocyanide is produced, which is partly soluble.

On settlement, the clear liquid was run to the drains or sewers, as it was then regarded as innocuous. No data, however, appear to exist as to the "oxygen absorbed" figure. In all probability the importance of this test had not in those days been disclosed.

The precipitate remained in the tank, pending the collection of a sufficient quantity to justify treatment. The precipitated cupreous sulphocyanide was well washed to remove impurities, and treated with an alkaline sulphhydrate, which decomposes into an alkaline sulphocyanide in solution, and a precipitate of sulphide

of copper. Advantage is again taken of the waste products by passing the foul gases from the saturator through a simple form of tower containing an alkali in solution, to produce an alkaline sulphhydrate for decomposing.

The sulphide of copper is reconverted into a copper salt by exposure to air and then dissolved in acid. In this way copper sulphate is regenerated, and the cupreous sulphocyanide can be converted into potassium cyanide by a direct process.

Details of the latter process were worked out by G. P. Lewis, who estimated in the days when it was propounded that, if the process were worked on a sufficiently large scale, it would yield a gross profit of about $3\frac{1}{2}$ d. per ton of coal carbonised, after making due allowances for the cost of materials, fuel, labour, and depreciation.

J. Radcliffe was granted a patent for a method of recovery of sulphocyanides from spent liquors in much the same way as that just outlined. The neutralised or slightly acid liquor is run into a tank to which is added a regulated amount of copper salt solution in a continuous manner. If the copper salt is in the cupric state, reduction to the cupreous state is effected by the addition of sulphurous acid. The cupreous sulphocyanide formed is separated by filter pressing, and by the aid of an alkali it is transformed into Cu_2O and marketable cyanogen compounds.

The elimination of any copper in the filtered liquor is effected by means of lime, or an alkali, or metallic iron. Spent liquor is then discharged in an innocuous state.

It is feared to-day that these processes are not commercially sound, although it is certain that the result of the treatment of the effluent liquors in the manner outlined would be beneficial.

How far the three methods which have been described can be combined so as to ensure a suitable effluent liquor for discharge is a matter for consideration and trial, but in any event it is certain that economic considerations are paramount in these days when the revenue from ammoniacal liquor has been sensibly reduced as contrasted with pre-War days.

CHAPTER XVI

THE USE OF CONCENTRATED AMMONIA LIQUOR IN THE PURIFICATION OF CRUDE COAL GAS

THE idea of the use of concentrated ammonia liquor for the purification of crude coal gas dates from 1817, when D. Wilson, of Dublin, took out a patent for purifying coal gas by means of the chemical action of *ammoniacal gas*.

Reference to the above circumstance is made in *A Historical Sketch of the Origin and Progress of Gas Lighting*, by Wm. Matthews (2nd edition, 1832). The method was again revived by Laming in 1860. He apparently purified his ammoniacal liquor by oxide of iron. In this way hydrogen sulphide was removed, and the purified liquor was fed to, and suitably distributed over, the packing medium of the scrubbers. It is recorded that this process was applied at the Bow Common Gas Works, and as far as one can ascertain it was successful in the removal of the bulk of the H_2S . Later, Hathaway introduced an improvement by which the furnace gases used in heating the retorts were utilised to effect the decomposition of the ammonium sulphide and the ammonium hydrosulphide of the gas liquor. The furnace gases from the retort settings were led to a scrubber through which ammoniacal liquor was passed. Records can be found which indicate that the process was a success so far as the elimination of H_2S was concerned; but it appears that this *modus operandi* was abandoned by reason of the loss of ammonia.

Subsequently, the late F. C. Hills discovered that by heating gas liquor to a temperature of about $80^{\circ}C$. he could drive off the CO_2 and H_2S without losing ammonia—thereby securing a caustic ammonia. The Hills process of liquid purification was introduced by Livesey, and put into operation at the Old Kent Road Gas Works many years ago, and a considerable portion of the gas produced at that works was for many years partially purified from H_2S and CO_2 . Later, Claus further adapted the process devised and worked by Hills, and installed plant and operated his process at the Windsor Street Gas Works of the Birmingham Corporation. Afterwards the Claus process was applied on a larger scale at the Belfast Corporation Gas Works.

In a contribution to the Institution of Gas Engineers, Stelfox described the operation of the process, furnished results, and, with commendable frankness, ascribed its failure to a so-called spectre—presumably a somewhat obscure loss of ammonia arising in the operation of the plant.

In more recent times liquid purification was introduced for a time by Holgate at the Halifax Gas Works, and by Davidson at Birmingham. It is not unlikely that success would have been achieved but for the outbreak of war. The foregoing is merely an epitomised historical survey.

THE PROCESS OF LIQUID PURIFICATION : OPERATIONS
AND CONSIDERATIONS INVOLVED

In general, the process of liquid purification contemplates the complete purification of gas by effecting the combination of the acid and alkaline impurities to form salts, which are readily soluble in water or weak ammoniacal solutions in circulation in a series of suitable towers.

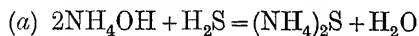
The process is one which is in operation continuously in all gas works every day. As, however, the acid impurities preponderate, ammonia must be added in adequate quantity to obtain the necessary combination.

The process involves four operations, as follows :

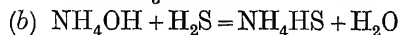
- (1) That in which the gas is purified.
- (2) That in which the dissociation of the ammonium compounds—such as ammonium sulphide, ammonium hydrosulphide, ammonium carbonate, and ammonium bicarbonate—is effected in order to remove the acid gases from the foul liquor.
- (3) That in which the purified liquor is distilled, with consequent production of caustic ammonia, which is used in the scrubbers for arresting the acid impurities.
- (4) That in which the acid gases arising from the dissociation of the ammoniacal liquor are utilised (a) by combustion for the production of SO_2 in connection with the manufacture of sulphuric acid, or (b) where the H_2S is recovered as Claus sulphur.

THEORETICAL PHASES OF THE PROCESS

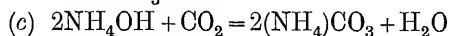
Before proceeding to a detailed consideration of the various operations necessitated in liquid purification, it will not be inexpedient to examine certain theoretical phases of the process in the light of the practical data available. Analyses reveal the presence of from 550 to 700 grains of H_2S per 100 cub. ft. of gas at the inlet to the purifiers ; or, assuming in all cases a make of 13,000 cub. ft. of gas per ton of coal carbonised, this represents 10.21 to 13 lb. of H_2S per ton of coal. Also 30 to 40 grains of CS_2 (more often 30 than 40) are present per 100 cub. ft. of gas at the inlet to the purifiers, or 0.56 to 0.74 lb. of CS_2 per ton of coal. Approximately 90 grains of HCN are present per 100 cub. ft. on the outlet of the last scrubber, or 1.67 lb. of HCN per ton of coal. Approximately 1800 grains of CO_2 per 100 cub. ft. are present in the gas on the inlet to the washers, and 350 grains of CO_2 per 100 cub. ft. are absorbed by the gas liquor in the washers and scrubbers. Thus 1450 grains of CO_2 per 100 cub. ft. must be removed in any process of liquid purification, or 30.6 lb. of CO_2 , 0.74 lb. of CS_2 , and 1.67 lb. of HCN have to be removed per ton of coal carbonised. To what extent will it be necessary to augment the circulation of ammonia in order to secure the combination of the acid impurities ? The reactions involved, viewed in the simplest form, are as follows :



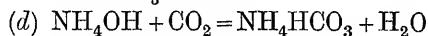
as NH_3 34.34.



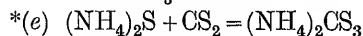
as NH_3 17.34.



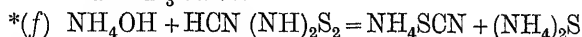
as NH_3 34.44.



as NH_3 17.44.



as NH_3 34.76.



as NH_3 17.27.

It is conceivable that other and more involved reactions than those marked by the asterisks (*) would occur; and it is equally conceivable that neither of the two reactions proceeds to completion. Ground Sicilian thirds sulphur could be introduced to the scrubbers in a suitable form in order to promote the formation of ammonium polysulphides, with a view to facilitating the removal of the HCN—one of the bugbears of the gas engineer, by reason of the destruction caused to plant, mains, and meters, etc. If it were deemed desirable to recover the hydrocyanic acid in such a form as to yield a product with a satisfactory monetary value, possibly an installation of two purifiers of adequate size, charged with suitable spent oxide, and worked on the basis of the Williams process—interposed between the tar extractor and the closed purification plant—would serve the purpose.

The liquor, consisting chiefly of ammonium sulphate and ammonium sulphocyanide, together with smaller quantities of free ammonium compounds and sulphate of iron, etc., resulting from the spraying of the spent oxide containing the arrested cyanide compounds, with water under pressure, could be collected and distilled with an alkali.

Alternatively the Davis-Neill process for the removal of cyanides could be adopted.

Proceeding now to the equations:

13 lb. of H_2S , based on reaction (a), will be removed by 13 lb. of NH_3 ; and

30.6 lb. of CO_2 , based on reaction (c), will be removed by 23.7 lb. of NH_3 .

0.74 lb. of CS_2 may conceivably be removed by 0.33 lb. of NH_3 (ammonia),

and 1.67 lb. of HCN may conceivably be removed by 1.05 lb. of NH_3 .

Altogether, 38.08 lb. of ammonia are required in circulation to effect the combination of the acid impurities in the crude coal gas.

The yield of ammonia per ton of coal carbonised may be based on the production of ammonium sulphate, which may be regarded as being equivalent to 30 lb., containing 25 per cent of ammonia per ton of coal carbonised. In other words, 7.5 lb. of HN_3 are recovered.

No attempt has been made to determine the extent to which the reactions

represented by equations (b) and (d) proceed, or to establish the factors which are likely to govern the limit to which the reactions as represented by equations (e) and (f) will proceed. These are doubtful points which demand research. In any case, the quantity of ammonia required in circulation is not materially affected by the reactions in question. Indeed, an allowance of 5 per cent would conceivably cover all four reactions. It is evident, therefore, from the data already furnished, that $\frac{38.08}{7.5} = 5.077$ times the quantity of NH_3 present in the gas is the minimum quantity required for ensuring the satisfactory purification of the crude coal gas by the process under review.

As the volume and composition of the gas vary from day to day—even from hour to hour—these variations can only be satisfactorily met by maintaining in stock in the scrubbers or circulating tanks (if used) an excess quantity of caustic ammonia, so as to provide against any conceivable variations or contingencies that may arise. The author would certainly recommend the circulation of at least seven times the quantity of ammonia available in the gas normally, so as to form the necessary reserve suggested.

DETAILS OF DESIGN AND ARRANGEMENT OF PLANT

Coming now to the design and arrangement of the plant for effecting the purification of gas by the method under review, and taking these *seriatim*, past experience would suggest that this portion of the plant should embody at least three essentials :

- (1) Adequate contact should be obtained between the crude coal gas and the re-circulated gaseous ammonia, in order to allow of the formation of the soluble ammonium compounds which are to be absorbed in the ammoniacal solutions and water being circulated. As the reactions involved necessitate time, as also does the absorption of the soluble ammonium compounds, the size of the vessels and the free space of the packing medium (assuming packed scrubbers are used) should be sufficiently large to allow of these desiderata being secured.
- (2) The gas to be purified should be freed as effectually as possible from tar and hydrocarbons, which are likely to accumulate and eventually prevent the satisfactory and efficient working of the packing medium used.
- (3) The relative merits and demerits should be carefully considered of (a) vertical scrubbers provided with suitable filling medium, which necessitate the installation of a series of bottom circulating tanks and pumps ; (b) horizontally disposed mechanical washer-scrubbers arranged in cascade form, in which a counter-current is established between the gas and re-circulated gaseous ammonia and the absorbing liquors and water.

Advantage could be taken in many cases of the varying levels of the site on which the projected plant is to be erected to dispense with the necessity for pumping the liquors to be circulated.

In this way loss of ammonia by spillage at the pump glands would be averted, as also that which is carried away with the displaced air from the storage tanks. These sources of loss in the past have contributed largely to the failure of liquid purification as a practical process. Indeed, in the writer's view, herein lies the possibility of relegating to the limbo of forgotten things the spectre of which Stelfox so eloquently spoke.

EXPERIMENTAL PLANT

In the lucid paper on the Claus ammonia process of gas purification read by Chas. Hunt to the Gas Institute in 1886, dimensions are given of the experimental plant he operated. Six scrubbers in all were used for the purification of the gas—five being 4 ft. square and 20 ft. high, filled with broken ganister bricks supported on cross angle-irons, while the sixth scrubber was 5 ft. in diameter, and 25 ft. high. The function of the latter apparatus was as a "finisher" or "catch," in order to arrest the last traces of impurity. The plant described was said to be equal to the purification of about 250,000 cub. ft. of gas per diem, a maximum of 300,000 cub. ft. having been obtained. Assuming the free space of the ganister brick packing per cubic foot to be 0.4, and the superficial free space per square foot to be 0.35 (not improbable figures), the velocity of the gas would be :

$$\frac{250,000}{24} \times \frac{1}{3600} = 2.9 \text{ cub. ft. per second.}$$

The superficial free space of the scrubbers Nos. 1 to 5 is 4 ft. by 4 ft. = 16 sq. ft. \times 35 per cent = 5.6 sq. ft.

$$(a) \text{ Velocity} = \frac{2.9}{5.6} = 0.52 \text{ ft. per second.}$$

Superficial free space of the final catch tower 5 ft. by 5 ft. by 0.78 = 19.5 sq. ft.

$$19.5 \text{ sq. ft.} \times 35 \text{ per cent} = 6.825 \text{ sq. ft.}$$

$$(b) \text{ Velocity} = \frac{2.9}{6.825} = 0.42 \text{ ft. per second.}$$

The total time-contact in the plant cited would be 251 seconds, or 4 min. 11 secs. From fairly extended experience of chemical reactions almost identical in character to those arising in liquid purification, the writer would be inclined to double the time-contact as the only certain way of securing efficient purification. As, however, such provision entails the duplication of the plant, and as any such duplication involves capital outlay, it is desirable to examine other arrangements of plant in the hope that the necessary time-contact, or something equivalent to it, may be attained without incurring twice the initial expenditure.

The plant under immediate consideration admits of reaction between the gaseous impurities and the liquid absorbents by film or surface-contact. However effective the wetted surface of the packing medium may be, or however absorbent or porous, or however tortuous or devious the path of the gases may be, those who have had

occasion to investigate the conditions affecting the absorption of soluble salts by liquids possessing considerable power of absorption know that (other things being equal) film-contact is not nearly so effective as the actual bubbling of the gaseous compounds to be absorbed through the absorbing medium, provided something approaching equable distribution of the gases through the available liquid space is secured.

The positive nature of the contact, where the actual forcing of the gases through the absorbing medium is effected, is strikingly demonstrated if one attempts to compare the absorption of the released gases from the dissociator of a concentrated ammonia

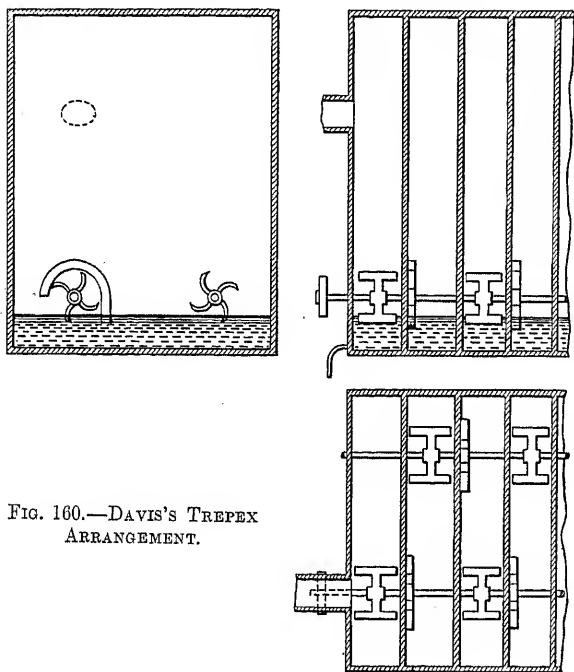


FIG. 160.—DAVIS'S TREPEX ARRANGEMENT.

liquor plant working under identical conditions: (a) In an absorber constituted of a series of trays, arranged in such a form as only to allow of film contact; and (b) an absorber where the gases have to be efficiently distributed through a definite number of seals of absorbing liquor. The size of the plant in example (a) will be more than twice the size of the plant in example (b). Only one drawback attends the latter. This is the extra resistance or back-pressure caused by actually forcing the gases through a series of seals as compared with the skin friction arising from film-contact. This leads the

author to the view that the scrubbers for admitting the combination of the acid and alkaline impurities and the absorption of the soluble ammonium salts so formed should embrace some positive arrangement of actual contact. Further, with a view to obviating the pumping of the absorbing liquor from tower to tower, and the provision of storage tanks at the foot of such towers, a series of horizontally disposed water-scrubbers of the Trepex type, designed to give positive contact, would be a decided advantage. If the latter plant embodied at the same time an arrangement for atomisation by the operation of a splasher, and if the various Trepex vessels were arranged in cascade form, so as to admit of the circulating liquors gravitating from vessel to vessel, no doubt a satisfactory solution would be afforded to the difficulties which it appears obsessed the minds and dogged the original workers of this process through the whole period of their experiments. A sketch of the Trepex apparatus is given in fig. 160.

but the arrangement illustrated does not embody any definite seal. Such provision can easily be made.

It is not necessary here to treat of the dissociation of gas liquor in the production of caustic ammonia. This aspect has been dealt with in Chapter XII. Neither is it necessary to proceed to a description of the treatment of the waste gases (hydrogen sulphide, carbon dioxide, and hydrogen cyanide, etc.) leaving the dissociator. Chapter XIV. has been assigned to a consideration of this phase.

ARRANGEMENT OF LIQUID PURIFICATION PLANT

To give an idea of the arrangement of plant, and the quantity of liquor to be dealt with, it will be assumed that liquid purification is proposed at a works carbonising 500 tons of coal per day.

To avoid circulating a large volume of concentrated ammonia liquor (caustic ammonia), and to reduce the cost of steam in the process of desulphurisation, etc., it will be appreciated that the higher the content of ammonia in the caustic liquor the better. The author's experience leads him to the view that caustic ammonia containing 6.3 per cent NH_3 can be advantageously used without risk of undue loss.

It may be counted that 42 gallons of gas liquor of 1.8 per cent NH_3 are produced per ton of coal carbonised. Thus, $500 \times 42 = 21,000$ gallons will represent the quantity of liquor produced per day, but the volume to be circulated will be :

$$\begin{aligned} & \frac{21,000 \text{ galls.} \times 7 \text{ times the daily quantity} \times 1.8 \text{ per cent } \text{NH}_3 \text{ of ammonia produced,}}{6.3 \text{ (the content of ammonia in the caustic ammonia to be circulated)}} \\ & \qquad \qquad \qquad = 42,000 \text{ gallons.} \end{aligned}$$

The author suggests that the decomposing plant should be operated at a pressure of, say, 30-40 lb. per square inch, in a steel dissociator. The idea underlying the use of a comparatively high pressure is based on an appreciation of the fact that at atmospheric pressure only a small range of temperature exists between the dissociation point of carbonate and sulphide of ammonium and the boiling-point of water, and as the solvent power of water for ammonia at these temperatures at ordinary atmospheric pressure is small, the process must be conducted slowly in large vessels, if loss is to be avoided.

If the process is conducted under considerable pressure, say, 30 lb. per square inch, as suggested, a greater quantity of caustic ammonia can be held in solution by the water while dissociation of the carbonate and sulphide is not sensibly affected ; thus stronger crude gas liquor can safely be employed.

Moreover, working with a pressure in the dissociator in question, it will be possible to feed the caustic ammonia to the finished still, without having recourse to pumping. This is a decided advantage.

FINISHING STILL

This should be of suitable size, and designed on the lines which have been indicated under "Ammonia Stills." Further, the dephlegmators for ensuring the production of gaseous ammonia free from water should be of adequate size, cooled by partially preheated liquor of a definite temperature, so as to avoid returning to the still a reflux of high ammonia content.

The condensers should be of efficient design, so as to avoid taxing the gas liquor plant with additional cooling. This is essential in view of the fact that gaseous ammonia has to be introduced to the inlet of the exhaustor after the condensers.

THE SUGGESTED LIQUID PURIFICATION PLANT

Fig. 161 shows a complete liquid purification plant as designed by the author, embodying most of the features outlined, all of which are directed to rendering the process comparable, economically, with that of dry purification as practised to-day.

METHOD OF WORKING THE PLANT

From the point of view of operation, the plant outlined in fig. 161 can be broadly divided into two parts; (a) the absorption, or purification portion of the plant, where the caustic ammonia arrests the impurities of the crude coal gas to form ammonium salts, which are dissolved in water and ammoniacal solution, and (b) the plant in which the desulphurisation and decarbonation of the resultant gas liquor and the production of caustic ammonia are undertaken.

One man per shift should be able to operate the first section of the plant, and a man and boy per shift should be able to work the second section of the plant. It will be necessary to carry at least a day's reserve of caustic ammonia, in the event of anything untoward happening to the plant comprising the second section.

Given satisfactory working, the production of caustic ammonia would practically coincide with that which is being circulated through the purification section.

Chemical control would be necessary, but it is assumed that this would not be greater than that needed in connection with solid purification as practised to-day.

TECHNICAL ASPECT

From the technical and chemical points of view there is no reason why concentrated ammonia liquor should not be successfully employed in the purification of crude coal gas. Indeed, a higher standard of purity of the final gas should be obtained than is possible with the present system of dry purification.

Whether it is wise to attempt to eliminate the whole of the impurities of the gas by liquid purification is a matter of doubt. The author's view is that the limit of expediency is reached at 90 per cent—the remaining 10 per cent of the impurities is best removed by dry purification. It should be remarked that the circulation

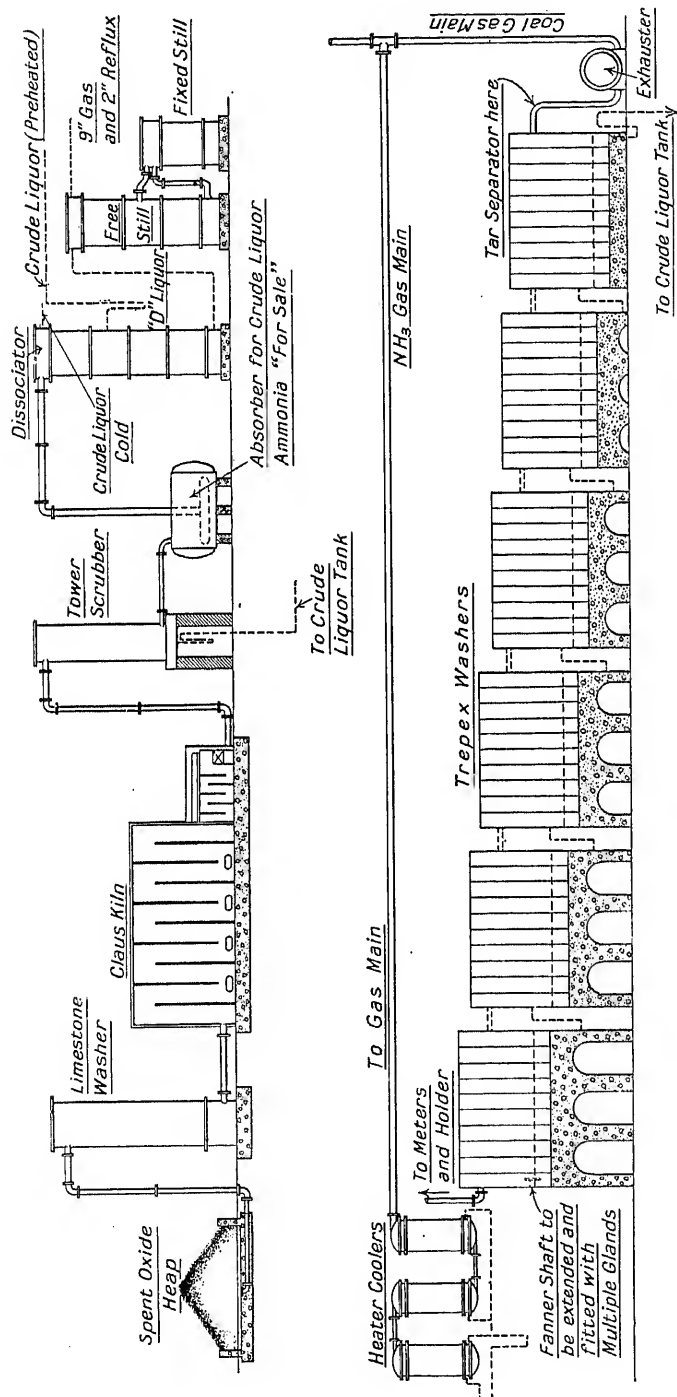


FIG. 161.—SKETCH OF LIQUID PURIFICATION PLANT.

of fairly strong ammoniacal solutions and the subsequent recovery of caustic ammonia is undertaken on a very large scale in the manufacture of soda ash by the ammonia-soda process, and there is no reason why concentrated ammonia liquor should not be used as the medium by which the elimination of the bulk of the impurities of crude coal gas is effected.

FINANCIAL ASPECT

Financial considerations have largely deterred gas undertakings from embarking on the wet process of purification during recent years. Figures do not appear to be available as representing the approximate cost of the wet purification process. The following cost sheet, prepared by the author, is intended to give a reasonably accurate idea of the cost entailed in the purification of crude coal gas by caustic ammonia.

TABLE 61

(1) Steam, 25 per cent (on 6.3 per cent NH_3) on 42,000 lb. of liquor, @ 4s. per ton of steam	£9 7 6
(2) Labour, 6 men and 3 boys @ 12s. and 9s. per shift respectively	4 19 0
(3) 10 cwt. of lime @ 35s. per ton	0 17 6
(4) Repairs and maintenance, say 5 per cent on capital	1 0 0
(5) Interest and depreciation on plant, £6000 at 10 per cent for 1 day (say)	2 0 0
	<hr/>
	£18 4 0

364s.
 500 tons of coal per day = 8.736d. as representing the purification of the coal gas from the carbonisation of one ton of coal.

The cost for dry purification of gas at the three largest London works for the year 1922, according to Field's Analysis, was 0.52d. per thousand cubic feet. Basing on thirteen thousand cubic feet per ton, this represents a cost of 6.76d. per ton of coal carbonised.

Of course, the former figure only covers the purification of 90 per cent of the crude coal gas, but it should be noted that no credit is given for the sulphur recovered. The latter figure constitutes the cost of the complete dry purification.

It is believed by some that one of the principal attractions of the liquid purification process disappeared with the introduction of the Gas Regulation Act of 1920, but the author does not subscribe to this view. There are many possibilities for the application of a simple process of liquid purification, and it would appear that coke oven works afford a peculiarly suitable field in this connection.

CHAPTER XVII

CARBONATION OF GAS LIQUOR, AND THE DISTILLATION OF THE CARBONATED LIQUOR

THE carbonation of gas liquor is a preliminary step in the manufacture of ammonium carbonate. Many methods have been proposed during the last few decades for the manufacture of this ammonium product. Some manufacturers prefer to use sulphate of ammonia and calcium carbonate (chalk) mixed in the proportion of one of the former to one and a half to two of the latter. This mixture is heated in an iron retort, and a little steam is used. The resulting volatilised product is ammonium carbonate.

Others prefer to distil pure liquor ammonia and produce ammonia gas with just the required humidity, to give with the gaseous carbon dioxide produced in a lime kiln, ammonium carbonate, when the constituents are brought together in a suitable subliming chamber. In other cases ammonium chloride is used with barium carbonate, ammonium carbonate being obtained as the primary product, and barium chloride as a secondary or by-product.

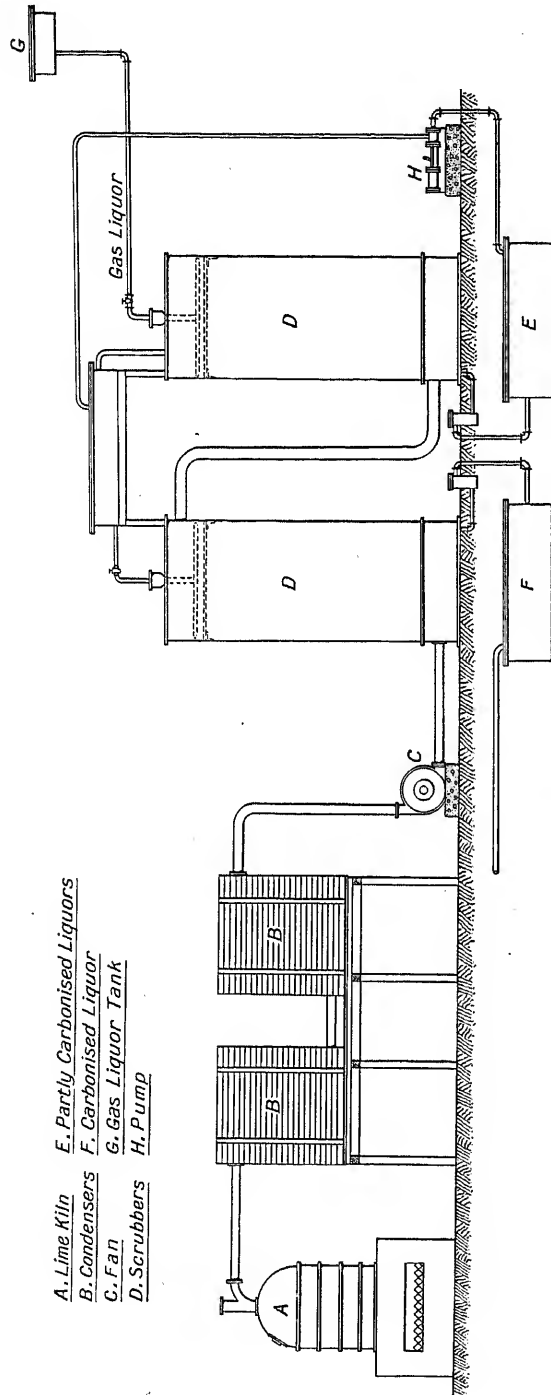
The method which has been responsible for the production of large quantities of ammonium carbonate in the past is one which involves the distillation of carbonated gas liquor. It is because this production has played such an important part in the past that a consideration is given here to the principles involved and the plant required.

PLANT REQUIRED FOR THE CARBONATION OF GAS LIQUOR

Fig. 162 shows a plant which consists (A) of a lime kiln, (B) two condensers built of chemical sheet lead, and (C) a fan and (D) two scrubbers, where the displacement of the hydrogen sulphide of the gas liquor takes place by carbon dioxide, with (H) a pump for circulating the partially carbonated liquor. Tanks (E), (F), and (G) are required respectively for partially carbonated liquor, carbonated liquor, and gas liquor.

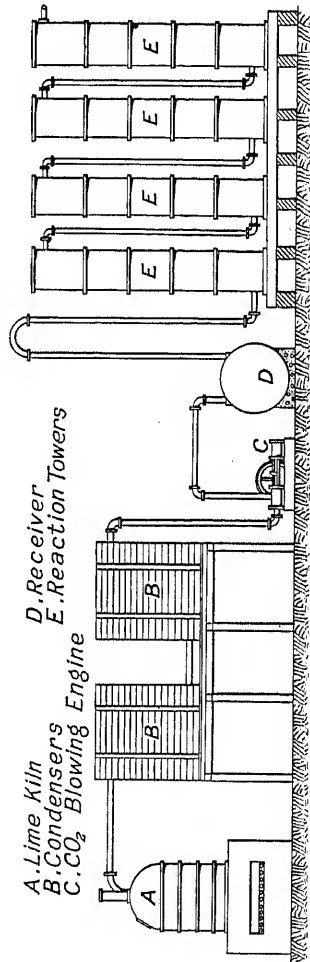
The plant indicated is sufficiently large to produce the carbonated gas liquor required for the manufacture of thirty tons of carbonate of ammonia per week.

The lime kiln shown is of an improvised type, yet it functions very satisfactorily. Pyrites kiln grate bars are used, and are set on angle when the kiln is under ordinary operation. The grate bars are periodically turned by a special key and the quicklime deposited, after which chalk and breeze are fed into the kiln at the top, and pending satisfactory ignition of the coke breeze, the gases are discharged from the stack pipe of the kiln, suitable adjustable dampers being provided for this purpose in the kiln stack pipe, and in the connection to the condensers. Given proper ignition of the fuel, then the carbon dioxide and steam generated are passed into the condensers, and are withdrawn by the fan and discharged through the scrubbers, down which partially carbonated liquor or gas liquor are uniformly distributed.



A. Lime Kiln
B. Condensers
C. Fan
D. Scrubbers
E. Partly Carbonised Liquors
F. Carbonised Liquor
G. Gas Liquor Tank
H. Pump

FIG. 162.—SKETCH OF PLANT FOR THE CARBONATION OF GAS LIQUOR.



A. Lime Kiln
B. Condensers
C. CO₂ Blowing Engine
D. Receiver
E. Reaction Towers

FIG. 163.—SKETCH OF BUBBLING APPARATUS FOR THE CARBONATION OF GAS LIQUOR.

Partially carbonated liquor is produced in the last scrubber, and this liquor is fed to the first scrubber, where carbonation is completed.

The feed of the scrubbers is maintained so as to give liquor practically free from H_2S . The hydrogen sulphide discharged from the last scrubber is sent forward for the production of sulphuric acid, or to a Claus sulphur kiln, or an oxide of iron purifier, as local circumstances may demand.

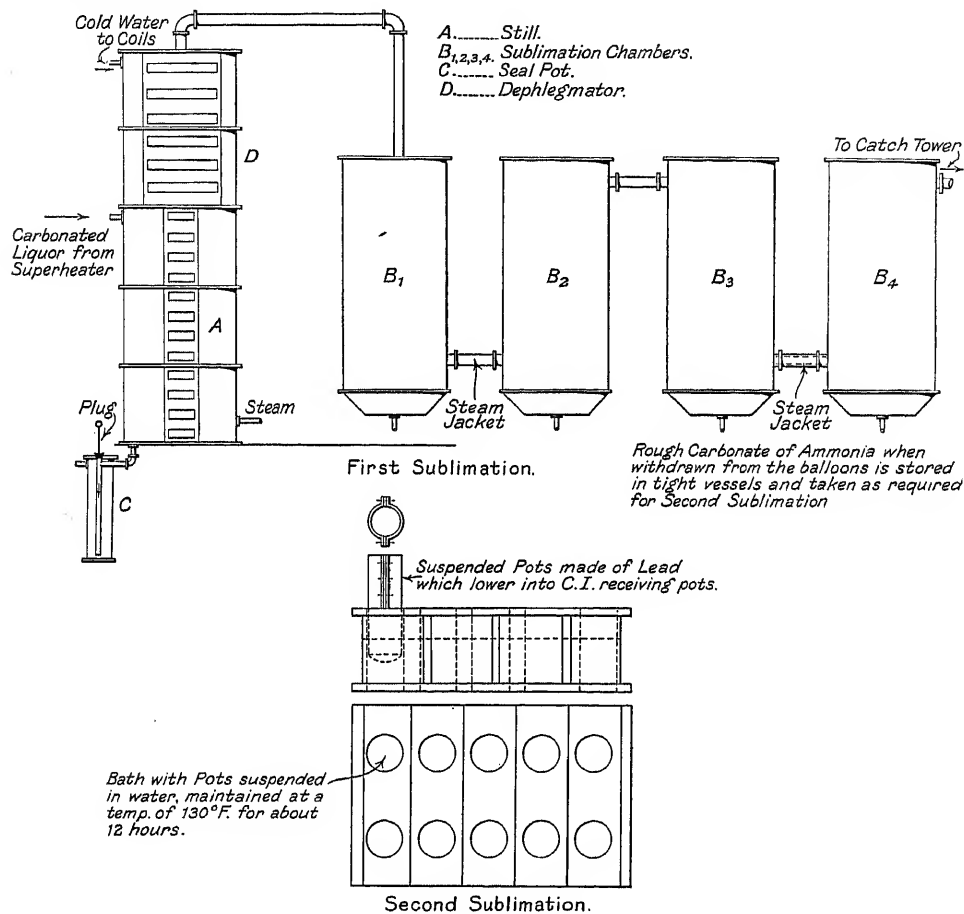


FIG. 164.—DISTILLATION PLANT FOR CARBONATED LIQUOR.

The kiln takes a charge of 12 to 14 cwt. of chalk every six hours, and approximately 20,000 gallons of gas liquor, 1.7 per cent NH_3 , are carbonated per day.

Fig. 163 represents a similar type of plant to the one just described, except that a more powerful blowing engine is used, and contact between the carbon dioxide and gas liquor is secured by bubbling through several perforated plates containing $\frac{3}{8}$ -inch diameter holes.

This type of carbonating tower functions very efficiently.

DISTILLATION OF THE CARBONATED LIQUOR

The carbonated gas liquor is distilled in an ordinary type of still, provided with suitably proportioned dephlegmators. The preheated liquor is fed to the still, and the ammonia, carbon dioxide, and steam are led to the first dephlegmator, which is cooled with cold carbonated gas liquor. Subsequently, the gases are led to the second dephlegmator, the cooling medium for which is water. It is important that the temperature of the gases emerging from the outlet of the second dephlegmator should be maintained at a uniform temperature of about 60°C . This temperature militates against possible blockage due to the formation of ammonium carbonate, and ensures the passage of such a quantity of steam with the ammonia and carbon dioxide as will produce a satisfactory quality of ammonium carbonate.

It is important in operating the still to ensure that the whole of the ammonium carbonate is decomposed before adding the milk of lime for the release of the fixed ammonia, otherwise trouble due to blockages in the still will arise by reason of the formation of calcium carbonate.

Fig. 164 shows diagrammatically the distillation plant for the carbonated liquor, as also the balloons in which the carbonate of ammonia is formed. Of course it will be appreciated that re-sublimation of the foregoing product in suitable vessels is necessary before the final commercial product is secured. An idea of the type of re-sublimation plant is afforded in fig. 164. The lead subliming pots made in semicircular form with easily detachable flanges allow of ready removal of the final product.

CHAPTER XVIII

CONSIDERATIONS GOVERNING THE PRICE OF GAS LIQUOR. SLIDING SCALE BASIS

Gas liquor is not sold on any uniform basis. In some cases a price per ton for certain strengths of liquor, delivered to railway tank wagons, or to tank barges at the gas works, is the method of sale; in other cases a price per unit of ammonia for certain strengths of liquor obtains; some prefer to consider offers for gas liquor based on a price per ton of coal carbonised, whereas a price per ounce per butt, or per butt of certain strengths of liquor, constitutes the basis of sale at other works.

Again, fixed prices apply in connection with certain arrangements, while sliding scale prices obtain in others.

The considerations governing the price of gas liquor are dependent on the nature of the contract whether a fixed price or a sliding scale one is to be adopted. Where a fixed price is desired, it is necessary to consider (a) the content of ammonia, and (b) the nature of the ammonia, whether free or fixed.

(a) *Content of Ammonia*.—This may vary from 1 to 4 per cent. Where it is desired to sell advantageously, it is obviously in the interests of all concerned that the gas liquor should be worked to as high a strength as possible. It is known that it is difficult in summer months to produce liquor with a much higher content of ammonia than 2 per cent, without by-passing 3 to 4 grains of ammonia per 100 cubic feet to the purifiers. There would be no great objection to this course but for the fact that an undue excess of ammonium compounds in the spent oxide affects prejudicially the consumption of nitric acid or nitrate of soda at the acid works, which is responsible for the combustion of the spent oxide in the manufacture of sulphuric acid. But there is no need why an acid catch should not be used, thus enabling gas works to produce gas liquor of 3 to 4 per cent strength without sending forward undue quantities of ammonia to the purifiers. This aspect will be the subject of consideration in the next volume.

It may, however, be assumed that it is a rational procedure to produce a strong liquor, thus lessening materially the costs of transport, and at the same time reducing certain of the manufacturing cost items—particularly steam. It is necessary to translate these considerations into figures to enable an adequate conception to be formed.

Table 62 shows the gas liquor to be transported per ton of sulphate of ammonia of 25.5 per cent NH_3 , with varying strengths of liquor, and it also reveals clearly the quantity of steam required per ton of sulphate of ammonia made with varying strengths of liquor.

When it is remembered that steam costs about five shillings per ton, even when generated from low-grade fuel, and that the railway rates for the conveyance of gas liquor may be as high as ten shillings per ton, it will be realised how important these items are.

TABLE 62

STEAM AND GAS LIQUOR REQUIRED PER TON OF SULPHATE OF AMMONIA
(25.5 PER CENT NH_3) MADE WITH VARYING STRENGTHS OF GAS LIQUOR

Per Cent NH_3 in Feed Liquor.	Lb. NH_3 per Ton Sulphate of Ammonia (25.5 per cent NH_3).	Tons Liquor per Ton of Sulphate of Ammonia (25.5 per cent NH_3).	Lb. Steam required per Ton of Sulphate of Ammonia (25.5 per cent NH_3).	Tons Steam required per Ton of Sulphate of Ammonia (25.5 per cent NH_3).	Per cent Steam on Gas Liquor distilled.
1.0	571.2	25.50	6438	2.88	11.6
1.5	571.2	17.00	4162	1.85	10.9
2.0	571.2	12.75	2963	1.32	10.4
2.5	571.2	10.20	2423	1.08	10.5
3.0	571.2	8.50	2031	0.91	10.7
3.5	571.2	7.28	1742	0.78	10.7
4.0	571.2	6.38	1581	0.71	11.1

(b) *Nature of the Ammonia*.—Manifestly, the nature of the ammonia must be considered, as it is much more expensive to work fixed than volatile ammonia. The author has, however, never seen figures submitted in this connection, but these are not difficult to compute. The items involved are (a) lime, (b) steam, (c) extra labour in the production of milk or cream of lime, and (d) labour for extra sludge removal.

TABLE 63

STATEMENT HAVING REFERENCE TO 5000 TONS OF GAS LIQUOR, THE TOTAL AMMONIA CONTENT OF WHICH IS 1.73 PER CENT, WITH FIXED AMMONIA CONTENTS OF 10, 20, AND 30 PER CENT RESPECTIVELY

Case.	A	B	C	D	E	F	G	H
A .	10	8.65	322.5	14.71	17.65	48,610	141	27.6
B .	20	17.30	322.5	29.42	35.30	97,220	282	55.2
C .	30	25.95	322.5	44.13	52.95	145,830	423	82.8

KEY

- A. Percentage fixed ammonia.
- B. Tons fixed ammonia in 5000 tons gas liquor (1.73 per cent total NH_3).
- C. Sulphate equivalent of total ammonia in 5000 tons of gas liquor (1.73 per cent NH_3).
- D. Theoretical lime equivalent of fixed ammonia (tons).
- E. Theoretical lime equivalent of fixed ammonia (tons), plus 20 per cent.
- F. Gallons water required to give 12° Tw. milk of lime.
- G. Tons steam required to raise water for milk of lime from 70° C. to 105° C.
- H. Tons sludge containing 60 per cent $\text{H}_2\text{O} + \text{CaSO}_4$ from 20 per cent of fixed ammonia and excess lime as calcium hydrate.

From the above figures it is possible to arrive at estimated cost sheets in the cases A, B, and C. These are appended separately (Table 64).

CONSIDERATIONS GOVERNING THE PRICE OF GAS LIQUOR 277

Take as an example 5000 tons of liquor at 1.73 per cent total ammonia, of which in Case A the fixed ammonia is 10 per cent of the total, in Case B 20 per cent of the total, and in Case C 30 per cent of the total.

It is desired to determine what the extra cost of manufacture will be per ton of sulphate of ammonia, 25.5 per cent NH_3 , given a 95 per cent working efficiency, and a lime consumption of (say) 20 per cent in excess of the theoretical. Table 63 furnishes full details from the point of view of lime, steam, water, and sludge, taking a 12° Tw. solution of milk of lime as the basis of the calculations.

TABLE 64

Cost Sheets

Case A

Lime—17.65 tons @ 35s.	£30 17 9
Steam—141 tons @ 5s.	35 5 0
Water—48,610 gallons @ 7d. per 1000 gallons	1 8 4
Sludge removal—27.6 tons @ 1s.	1 7 7
Labour (milk of lime preparation) (about)	4 0 0
	<hr/>
	£72 18 8

$$\frac{£72 : 18 : 8}{322.5 \text{ tons of sulphate of ammonia}} = 4\text{s. } 6\text{d. per ton.}$$

Case B

Lime—35.30 tons @ 35s.	£61 15 6
Steam—282 tons @ 5s.	70 10 0
Water—97,220 gallons @ 7d. per 1000 gallons	2 16 8
Sludge removal—55.2 tons @ 1s.	2 15 2
Labour (milk of lime preparation) (about)	8 0 0
	<hr/>
	£145 17 4

$$\frac{£145 : 17 : 4}{322.5 \text{ tons of sulphate of ammonia}} = 9\text{s. per ton.}$$

Case C

Lime—52.95 tons @ 35s.	£92 13 3
Steam—423 tons @ 5s.	105 15 0
Water—145,830 gallons @ 7d. per 1000 gallons	4 5 0
Sludge removal—82.8 tons @ 1s.	4 2 9
Labour (milk of lime preparation) (about)	12 0 0
	<hr/>
	£218 16 0

$$\frac{£218 : 16\text{s.}}{322.5 \text{ tons of sulphate of ammonia}} = 13\text{s. } 6\text{d. per ton.}$$

It will be seen from the foregoing cost sheets that 4s. 6d. per ton is involved in dealing with liquor having 10 per cent of the total ammonia as fixed ammonia, 9s. per ton in the case of 20 per cent of the total ammonia as fixed ammonia, and 13s. 6d. per ton in the case of 30 per cent of the total ammonia as fixed ammonia.

Apart from the latter considerations, the question of the unit price of ammonia must be considered.

In determining the unit price of ammonia the chemical manufacturer who is responsible for the offer or tender invariably takes his manufacturing costs into account, and assumes a certain nett realisation for the sulphate of ammonia. Of course there is an element of speculation where it is proposed to quote a fixed price. Manufacturing costs may increase or decrease; similarly, the nett selling price for the period covered by the contract has to be conjectured. Hence, in these circumstances, the manufacturer, with every desire to be fair, must cover himself. It will be appreciated that where a fixed price is the basis of the purchase of gas liquor, a contract, generally speaking, is for a short tenure—say, six or twelve months. Rarely do these contracts extend over a period of more than two years.

A decade ago contracts of the character in question usually contemplated a price of 4s. 6d. per unit of ammonia, or 9s. 5d. per ton for gas liquor of 10 oz. strength—2.17 per cent NH_3 —delivered to buyer's tank wagons, where the railway rate paid by the chemical manufacturer was 4s. 4d. per ton. Costs of production of sulphate of ammonia, 24 per cent NH_3 , were £4 per ton, and the nett selling price, f.o.b. Hull, for good grey material was £13 : 16 : 8 per ton.

It will be interesting to examine the foregoing particulars to determine what was the probable profit to the chemical manufacturer, and incidentally to ascertain whether the gas undertaking was securing a reasonable price for its production of liquor.

DATA

TABLE 65

1 ton of sulphate of ammonia, 24 per cent	0.24 ton NH_3
1 ton of gas liquor, 10 oz., 5° Tw.	0.0217 ton NH_3
$\therefore \frac{0.24}{0.0217} = 11.06$ tons of gas liquor required for the production of one ton of sulphate of ammonia.	

Assume in this case a $92\frac{1}{2}$ per cent efficiency :

$$\frac{100 \times 11.06}{92.5} = 11.95 \text{—say, 12 tons of gas liquor required.}$$

A balance sheet can now be prepared thus :

TABLE 66
Balance Sheet

<i>Dr.</i>		<i>Cr.</i>
To cost of manufacture . . .	£4 0 0	By nett selling price for 1 ton
Cost of gas liquor, 12 tons @		of sulphate of ammonia,
9s. 5d.	5 13 0	good grey, 24 per cent
Railway carriage, 12 tons @		NH ₃
4s. 4d. per ton	2 12 0	£13 16 8
Tank hire, 1 week @ 1s.	0 12 0	
Balance (profit)	0 19 8	
	<hr/>	
	£13 16 8	<hr/>
		£13 16 8

It is seen from the above that the extent of the profit is about 1s. 7d. per ton of gas liquor, or 17 per cent. Where a risk of falling prices has to be run, and an advance of manufacturing costs is possible, one can scarcely cavil at such a profit.

SLIDING SCALE BASIS

It must, however, be manifest that the most satisfactory basis of arrangement alike to the chemical manufacturer as to the gas undertaking is one which contemplates a sliding scale, so as to reduce the element of speculation to a minimum. In this case the manufacturer is in a position to pay much better prices for the gas liquor to be supplied, and the seller has the satisfaction of knowing that the arrangement is calculated to admit of best possible terms being paid for his production of liquor.

Now a sliding scale may be governed by several considerations. It may contemplate (a) the strength of the liquor supplied, (b) the nett price realised for the sale of sulphate of ammonia, and it may, and indeed often does, contemplate (c) the cost of manufacture of the sulphate or other ammonium product. It must also contemplate (d) a certain production efficiency.

It may provide a sliding scale price for varying strengths of liquor, irrespective of the foregoing slide. It will be appreciated that gas liquor of different strengths possesses different values to the chemical manufacturer, and if the conditions at the gas works prevent the supply of a definite and uniform strength of liquor throughout the currency of the contract, then it often saves time and correspondence if the contract provides a price for the various strengths of liquor likely to be supplied.

In connection with any such sliding scale contract, it will be realised that what has been said anent (a) the strength of the liquor supplied when dealing, in the former part of this chapter, with the question of a fixed price is equally applicable to a sliding scale arrangement.

(b) *Nett Price realised for the Sale of Sulphate of Ammonia.*—At one time it

was the practice to take the figures quoted in the *Gas Journal* week by week, and to deduct $2\frac{1}{2}$ per cent discount and 1 per cent commission, and the cost of lighterage or railway carriage from the sulphate works to f.o.b. the port at which the product was delivered.

In this way a satisfactory figure was reached. Recently, however, the practice has been to call for a certificate from a competent authority representing the nett sulphate price at port, and to make the necessary deduction in respect of freightage or carriage as the case may be.

In any case, it should not be a difficult matter to secure an authentic figure in this connection which will be accepted without question both by the buyer and the seller.

(c) *The Cost of Manufacture of Sulphate of Ammonia, or other Ammonium Product.*—It is usual when submitting a tender on the basis of a sliding scale contract to name a figure representing the cost of manufacture, and to state that this figure will vary half-yearly or yearly, as the case may be, according to wages, price of fuel, raw materials, and repairs and maintenance expenses, etc. By furnishing a figure when tendering the seller has an opportunity of determining not only whether the costs of manufacture in question are reasonable, but whether he is allying himself with an efficient or indifferent firm. With an indication at the outset of the contract as to the manufacturing costs, there is little difficulty in reaching an agreement as regards the subsequent adjustment, if any.

In general, these adjusted figures will not vary very widely, although they may constitute a slight advance or reduction on the originally indicated manufacturing costs.

(d) *Production Efficiency.*—What over-all efficiency can the manufacturer safely base upon, and what is reasonable from the point of view of the seller of the gas liquor? A 95 per cent over-all efficiency should be possible in the manufacture of sulphate of ammonia, and a 93 per cent efficiency with liquor ammonia, muriate of ammonia, and carbonate of ammonia.

Usually, however, an over-all efficiency figure of $92\frac{1}{2}$ per cent is taken as the basis of sliding scale contracts, and, generally speaking, this may be regarded as a fair average.

It will be seen that one is now in possession of the essential details to admit of a contract on a sliding scale basis being arrived at.

Perhaps it will be desirable to translate the matter into figures in order to make the position explicit.

Two types of sliding scale contracts will be considered: (A) one where the strength of the gas liquor is fixed, and the other (B) where the strength of the gas liquor varies between certain well-defined limits.

Example (A) has reference to a gas works which undertakes to supply gas liquor of 12 oz. (6° Tw.) strength, delivered to buyer's railway tank wagons f.o.r. seller's sidings. The railway rate to buyer's works is 3s. 3d. per ton.

DATA

TABLE 67

- (a) 12 oz. liquor = 2.604 per cent NH_3 , or in one ton there is 0.02604 ton of ammonia.
- (b) One ton of sulphate of ammonia (25.5 per cent) contains 0.255 ton of ammonia.
- (c) $\frac{0.255}{0.02604} = 9.792$ tons.
- (d) Basing on 92.5 per cent over-all efficiency,

$$\frac{9.792 \times 100}{92.5} = 10.586$$
tons of gas liquor required in the production of one ton of sulphate of ammonia (25.5 per cent NH_3).
- (e) Manufacturing costs, including tank hire, £8 per ton.
- (f) Nett works selling price on which the scale is based, £14 : 10s. per ton.
- (g) The profit which the chemical manufacturer contemplates is $12\frac{1}{2}$ per cent on the cost of production, i.e. £1, making the manufacturing cost plus profit into £9 per ton.
- (h) There is therefore available for appropriation in respect of liquor involved in the production of one ton of sulphate :

Nett works selling price	£14 10 0
Cost of manufacture, plus profit	9 0 0
Difference	£5 10 0
Carriage, 10.586 tons @ 3s. 3d. per ton = (say)	1 14 6
Balance	<u>£3 15 6</u>

- (i) $\frac{75.5 \text{ shillings}}{10.586 \text{ tons of gas liquor}} = (\text{say}) 7\text{s. } 1\text{d. per ton.}$

- (j) $\frac{10\text{s. per ton advance or decrease in price of sulphate of ammonia}}{10.586 \text{ tons of gas liquor}} = 11\frac{1}{4}\text{d. per ton.}$

From the foregoing it will be seen that a tender could be submitted in the following form :

" We have pleasure in quoting you for your surplus production of ammoniacal liquor, estimated at 5000 tons per annum, on the undermentioned basis :

- "PERIOD OF CONTRACT. Three years, commencing from July 1 next.
- "STRENGTH OF LIQUOR. 12 oz., by the distillation test.
- "DELIVERY. To be to buyer's railway tank wagons, at seller's sidings.
- "WEIGHING AND SAMPLING. To be at seller's works at time of loading, buyer to be represented thereat if he so desires.
- "PRICE. The following sliding scale prices to obtain for the first year's deliveries. For each subsequent year, the nett manufacturing cost plus profit shall be reviewed and the prices per ton adjusted *pro rata*.

"Sliding Scale for First Year"

Nett Selling Price for Sulphate of Ammonia containing 25.5 per cent NH_3 .							Price per Ton of 12 oz. Liquor.	
£18	0	0	£0	13 7 $\frac{3}{4}$
17	10	0	0	12 8 $\frac{1}{2}$
17	0	0	0	11 9 $\frac{1}{4}$
16	10	0	0	10 10
16	0	0	0	9 10 $\frac{3}{4}$
15	10	0	0	8 11 $\frac{1}{2}$
15	0	0	0	8 0 $\frac{1}{4}$
14	10	0	0	7 1
14	0	0	0	6 1 $\frac{3}{4}$
13	10	0	0	5 2 $\frac{1}{2}$
13	0	0	0	4 3 $\frac{1}{4}$

- "DETERMINATION OF NETT SELLING PRICE. A certificate shall be issued by a competent authority each quarter, declaring the nett selling price for the preceding three months.
- "PAYMENT. Nett cash quarterly."

Consideration will now be given to Example B, where it is desired not only to cover conditions such as the foregoing, but where it is necessary to quote a differential price for several strengths of liquor varying from 10 to 13 oz., owing to the inability of the gas undertaking to guarantee a uniform quality of liquor throughout the year.

It will be assumed that for the most part the gas undertaking will deliver 12 oz. liquor, but that in the summer months gas liquor as low as 10 oz. strength will be supplied. In order to show the application of differential prices it will also be assumed that during the winter months occasional tank loads of gas liquor of 12.5 oz. and 13 oz. strength are sent to the chemical works.

Now the factors which affect the price of gas liquor when strengths below or above the standard are sent along are (a) steam, (b) carriage of liquor, (c) tank hire, and (d) to some extent wages and overhead charges.

It is not so easy to say to what extent the still capacity is affected when a stronger or a weaker liquor than the standard is distilled. The author's experience is that under practical conditions a distillation unit intended to deal with (say) 30,000 gallons of liquor per day of 8 oz. strength will treat satisfactorily the same volume of 10 oz. liquor, but that a disproportionate loss of ammonia occurs if an attempt is made to distil stronger liquor than 10 oz.

As a result of the examination of many cost sheets, not confined to one works alone, but affecting several works, the author has reached the conclusion that certain items in the cost of manufacture of sulphate of ammonia, such as sulphuric acid, bags, etc., remain the same, irrespective of the volume of liquor used per ton of sulphate made, but others—steam, wages, carriage on liquor, etc.—vary according to the strength of the liquor treated. The ratio of the items which vary according to the volume of the liquor invariably represents 40 per cent of the total cost of manufacture. Reference to an earlier part of this chapter will show that the price to be paid for 12 oz. liquor, when the nett selling price of sulphate of ammonia is £14:10s. per ton, is 7s. 1d. per ton.

The method of procedure to arrive at the sliding scale is as follows:

The cost of manufacture is £8 per ton, and the cost of carriage is £1:14:6, giving a figure of £9:14:6. Of this amount:

40 per cent	is	£3:17:10,	representing	the	variable	items	; and
60	„	£5:16:8,	„	„	fixed	items.	

The next step is to determine the fluctuating cost of manufacture for varying strengths of liquor. Such fluctuating cost is in inverse proportion to the strength of the liquor. Having arrived at what the variable cost of manufacture is for each particular strength of liquor, this cost, which may be regarded as x , must be added to the fixed cost of manufacture, giving the total cost, or fixed cost plus x . Table 68 furnishes these data.

The next procedure is to deduct the cost of manufacture, plus £1 per ton profit, peculiar to each strength of liquor, from the nett selling price of sulphate of ammonia. A balance is arrived at in each case which is divided by the number of tons of that particular strength liquor in respect of which it is desired to determine the price per ton. By way of example, take 10 oz. liquor: it will be seen from Table 68 that the balance available for division into the weight of liquor involved is £3 when the nett selling price of sulphate of ammonia is £14:10s.

As 12.7 tons of 10 oz. liquor are required in the manufacture of one ton of sulphate of ammonia, given a $92\frac{1}{2}$ per cent efficiency, then

$$\frac{60\text{s.}}{12.7 \text{ tons}} = 4\text{s. } 9\text{d. per ton as the value of the gas liquor.}$$

TABLE 68

										Cost of Manufacture according to Strength of the Liquor.						
				Variable Item.			Fixed Item.									
<i>s.</i>				<i>s.</i>	£	<i>s.</i>	<i>d.</i>		£	<i>s.</i>	<i>d.</i>		£	<i>s.</i>	<i>d.</i>	
77.8	×	$\frac{12}{10}$	=	93.36	=	4	13	4	+	5	16	8	=	10	10	0
77.8	×	$\frac{12}{10.5}$	=	88.93	=	4	8	11	+	5	16	8	=	10	5	7
77.8	×	$\frac{12}{11}$	=	84.88	=	4	4	11	+	5	16	8	=	10	1	7
77.8	×	$\frac{12}{11.5}$	=	81.20	=	4	1	2	+	5	16	8	=	9	17	10
77.8	×	$\frac{12}{12}$	=	77.80	=	3	17	10	+	5	16	8	=	9	14	6
77.8	×	$\frac{12}{12.5}$	=	74.69	=	3	14	8	+	5	16	8	=	9	11	4
77.8	×	$\frac{12}{13}$	=	71.87	=	3	11	10	+	5	16	8	=	9	8	6

If the nett selling price of sulphate of ammonia is £18, then £6 : 10s. is available for distribution into the 12.7 tons of liquor involved, thus giving a figure of (say) 10s. 3½d. per ton.

If another case is considered where the liquor is higher than the standard of 12 oz., the following calculations are involved :

Take 13 oz. liquor : It will be seen in this case that the variable items of cost only amount to £3 : 11 : 10 per ton, giving a total cost of manufacture of £9 : 8 : 6, which, with £1 per ton profit, aggregates to £10 : 8 : 6. If the nett selling price of sulphate of ammonia is taken at £14 : 10s. per ton, the amount available for distribution is £4 : 1 : 6. This amount, divided by the number of tons involved (9.77),

$$= \frac{81.5s.}{9.77 \text{ tons}} = 8s. 4d. \text{ per ton.}$$

If the nett selling price of sulphate of ammonia were £18 per ton, then the amount available for distribution would be £7 : 11 : 6, and this divided by the number of tons involved (9.77) gives (say) 15s. 5¼d. per ton.

On the basis of calculations of this character the following sliding scale is arrived at :

TABLE 69

Nett Selling Price for Sulphate of Ammonia containing 25·5 per cent NH ₃ .	Price per Ton of Gas Liquor.													
	10 oz.		10·5 oz.		11 oz.		11·5 oz.		12 oz.		12·5 oz.		13 oz.	
£ s. d.	s.	d.	s.	d.	s.	d.	s.	d.	s.	d.	s.	d.	s.	d.
18 0 0	10	3½	11	2	12	0½	12	9¼	13	7¾	14	7¼	15	5¾
17 10 0	9	6	10	4	11	2	11	10½	12	8½	13	7½	14	5½
17 0 0	8	8½	9	6	10	3½	10	11¾	11	9¼	12	7¾	13	5¼
16 10 0	7	11	8	8	9	5	10	1	10	10	11	8	12	5
16 0 0	7	1½	7	10	8	6½	9	2¼	9	10¾	10	8¼	11	4¾
15 10 0	6	4	7	0	7	8	8	3½	8	11½	9	8½	10	4½
14 10 0	4	9	5	4	5	11	6	6	7	1	7	9	8	4
14 0 0	3	11½	4	6	5	0½	5	7¼	6	1¾	6	9¼	7	3¾
13 10 0	3	2	3	8	4	2	4	8½	5	2½	5	9½	6	3½
13 0 0	2	4½	2	10	3	3½	3	9¾	4	3¼	4	9¾	5	3¼

There is only one comment which need be made concerning the above sliding scale. It refers to the profit of the chemical manufacturer, which remains stationary, irrespective of the nett selling price of sulphate of ammonia. It may be suggested that this is hardly fair to the chemical manufacturer. On the other hand, if the plea is urged that the buyer of gas liquor should share the prosperity of the gas undertaking when the nett selling price of sulphate of ammonia advances, it will be agreed that it is only common justice to expect the chemical manufacturer to sacrifice some of his profit in times of adversity when the gas undertaking suffers by reason of a fall in the selling price of sulphate of ammonia below that contemplated when the sliding scale was devised.

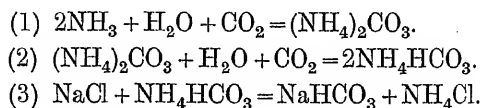
In all these matters a reasonable equipoise must be maintained. No sliding scale basis or other scheme can possibly legislate for every contingency, but a sliding scale of the above character is calculated to reduce the elements of speculation or contingent possibilities (call them what you will) to a minimum.

This chapter does not profess to exhaust all the possible sliding scale schemes for the sale or purchase of gas liquor, but it is hoped that sufficient has been said to assist in a general appreciation of the underlying considerations involved, as indeed in the steps which must be taken in the determination of the figures constituting a sliding scale.

CHAPTER XIX

AMMONIA STILLS OF THE AMMONIA-SODA PROCESS

THE production of ammonia-soda involves the passage of carbon dioxide through brine saturated with ammonia, when the following reactions occur in a consecutive manner :



The brine is pumped direct to the ammonia saturators, and does not usually undergo any preliminary purification. The saturators, as will be seen from fig. 165, take the form of a tower, in connection with which special serrated distributing devices are used for the ammonia. These devices are sealed to a pre-determined depth in brine solution, which overflows from tray to tray. As has been remarked in Chapter XII., concentrated ammonia liquor containing about 0.5 per cent of H_2S , on the basis of 25 per cent NH_3 , is used to restore the ammonia lost in the process. The temperature of the brine solution must be maintained below 60°C . by suitable means. The gas emerging from the saturators is passed through a second tower, or washer, to arrest the ammonia. The final tower is coupled to a vacuum pump, so as to reduce the pressure against which the ammonia issuing from the stills has to work.

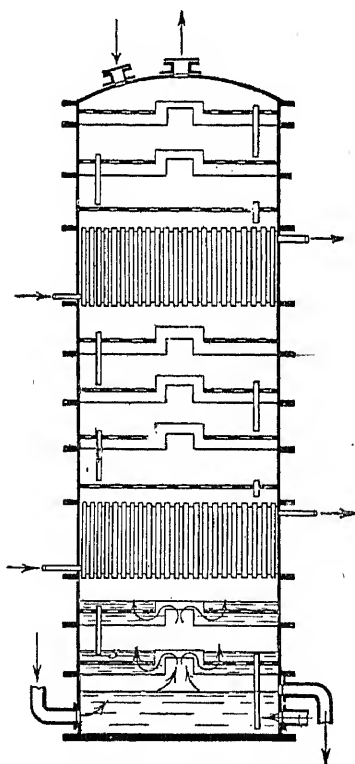


FIG. 165.—SATURATOR FOR AMMONIATING BRINE.

Due to the presence of sodium and ammonium bicarbonate in the mother liquor from the carbonating tower, it will be found that carbon dioxide is always associated with the gaseous ammonia from the stills. The liquid leaving the ammonia saturators usually has a composition of the following order :

	NaCl.	NH ₃ .	CO ₂ .
Grams per litre	260	80	45

To remove the suspended carbonates of calcium and magnesium arising from the impurities in the brine, the liquid leaving the saturators is passed to decantation tanks. A clear supernatant liquor of tolerably constant composition is produced, and this is cooled to 30°C . to prevent loss of ammonia, and is then fed to the carbonating

apparatus. Meanwhile, the mud recovered in the decantation tanks is treated for the recovery of ammonia.

Carbon dioxide is pumped through the clear supernatant liquor, which is distributed over a Solvay tower, in the lower portion of which Cogswell coolers—a multitubular form of cooler, consisting of a number of mild steel pipes placed horizontally, and through which water flows—are expanded into tube plates.

Carbonation usually proceeds in two stages, giving rise (1) to the production of ammonium carbonate, and (2) to the formation of ammonium bicarbonate, and the deposition of sodium bicarbonate. It is the practice to carry out each stage in a separate tower, and in the second apparatus a definite temperature of about 30° C. is maintained, so as to produce bicarbonate of the proper consistency.

Owing to the formation of hard crusts of bicarbonate in the second tower, which create blockages, it is necessary to reverse the order of working the towers, and in this way the crusts are dissolved.

The carbon dioxide used in the process of carbonation is obtained from two sources, either (a) lime-kilns, or (b) calcination of bicarbonate in the production of soda ash.

The CO₂ content of lime-kiln gas varies, of course, with the working, but reaches as high as 40 per cent. The calciner gas varies in CO₂ content from 90 to 98 per cent. In practice, a mixture of the two is favoured for the carbonation of the ammoniacal brine. In this way an average of about 53 per cent CO₂ is secured. The mother liquor emerging from the carbonating process along with the magma of bicarbonate undergoes filtration in a specially designed rotary filter, embracing a perforated drum covered with special filter cloth. A portion of the filter cloth dips into the dish containing the semi-solid, and a suction is applied from inside. Thus a layer of sodium bicarbonate collects on the filter cloth. This is washed with water to remove traces of ammonia, and is subsequently dislodged by knives as the cylinder rotates.

During the washing of the cake the mother liquor is diluted, and contamination with sodium bicarbonate occurs. The mother liquor contains practically all the ammonia originally present in the ammoniacal brine.

It is to the stills used in the dissociation of the ammonia from the mother liquor in question that this chapter is especially assigned. This mother liquor has the following approximate analysis :

1.8	per cent of volatile ammonia, and
6.0	,, of fixed ammonia, largely as NH ₄ Cl ;
<hr/>	
=7.8	,, total NH ₃ .
<hr/>	

The principle followed in the treatment of the ammoniacal mother liquor is much the same as that pursued in the distillation of gas liquor. The stages of the distillation or dissociation are broadly divided into three. Initially, the volatile ammonia is dissociated, after which the liquor containing the fixed ammonia is mixed

with cream of lime in a special rotary mixer, and subsequently the limed liquor is distilled. At all events, this is the procedure with the latest type of apparatus.

Instead of a steam and ammonia ratio of 85-80 : 10-15, which obtains in the manufacture of sulphate of ammonia, a fairly pure and dry mixture of carbon dioxide and ammonia is evolved in the distillation of the ammonium chloride liquors. The latter gases are led to saturators for the preparation of ammoniated brine.

STILLS

Fig. 166 shows a section of a Mond still, somewhat analogous to one in use at an American factory dealing with the ammoniacal mother liquor in connection with a plant producing 100 tons of soda ash per day of 24 hours. The height of the American still, which is made of a steel-iron, is 91 ft., and its diameter is 10 ft. It comprises thirty-three circular compartments—eleven of which at the foot perform the distillation proper, and the remainder the preheating and dephlegmation. Ammonium chloride solution is admitted at the top, and milk of lime by two inlets in the tenth and eleventh compartments. The overflows are not arranged internally, as shown in fig. 166, but externally, with lateral joints. Facility of cleaning is the object in view. The orifices for the passage of the gases are 24 in. wide, and the caps covering them have a lower diameter of 7 ft.

But the ammonia-soda industry has not been without a boiler type of still, and in this respect there is an analogy with the plant used in the distillation of gas liquor. Fig. 167, first and second views, shows the ammonia stills consisting of four boilers (A); the distributor (B); the distilling column (C); the cooler (D); the ammonia absorber (E); the vacuum pump (F), and the mother liquor tank (G). The boilers are provided with a conical bottom, reserved for distillation of the liquor with lime. The third view shows the equipment of such a boiler, which has two pipes connecting it with distributor (B)—pipe *n* for the inlet and pipe *o* for the outlet of the gas. The latter terminates at the top of the boiler; the former (*n*) is a sort of dip pipe, terminating within 4 in. of the conical bottom. A sieve is fixed about the centre of the straight portion of the

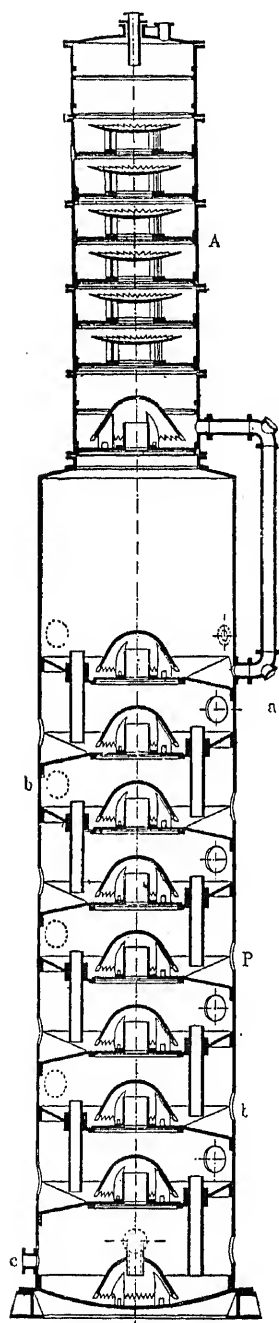


FIG. 166.—AMMONIA STILLS—
AMMONIA-SODA PROCESS.

boiler. Prior to entering the boiler, a branch pipe *p* connects pipe *n* with a blow-off pipe *r* for spent liquor; *p* can be closed by the bell valve. The cock *r* of the discharge pipe is placed behind the junction of the pipe. The discharge pipe dips into the boiler almost as deeply as the steam pipe. Hence, during distillation, when *x* is opened, steam flows through *p* and *r* into the boiler, and keeps the discharge pipe clean. To avoid the escape of liquor and steam from the discharge cock, it is secured by a stop flange behind the cock.

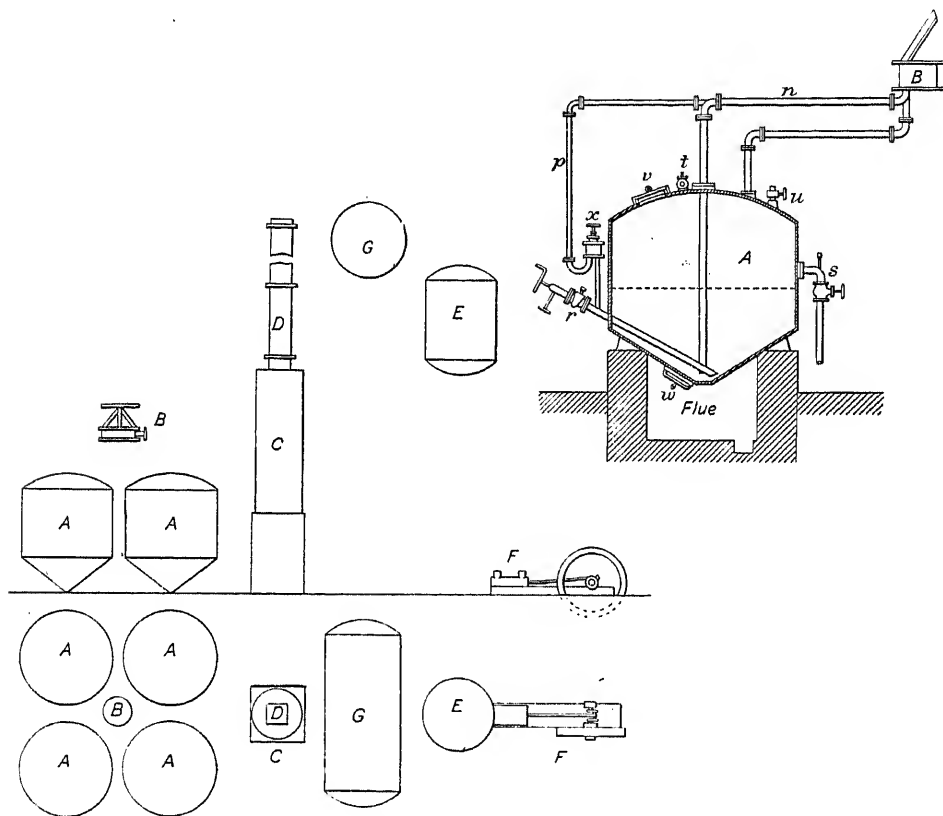


FIG. 167.—AMMONIA STILLS—FOUR VIEWS OF THE AMMONIA-SODA PROCESS.

The waste liquor which is carried away by a pipe (not shown) serves for preheating the boiler feed water prior to leaving the works.

The steam space of the boilers is connected with that of the column, so as to put this in pressure equilibrium during the charging of the ammoniacal mother liquor. The feed pipe can be shut off by a valve *t*, which it is desirable to provide with a stop flange. Cock *u* serves for the introduction of milk of lime.

The boilers are provided with manholes, pressure gauges, and several sampling cocks, which also serve for determining the height of the liquor. The boiler is carried

on brickwork by suitable brackets riveted to the conical portion in such a way as to admit of the bottom of the boiler being accessible.

The function of the distributor B is to cut off one of the boilers from the steam

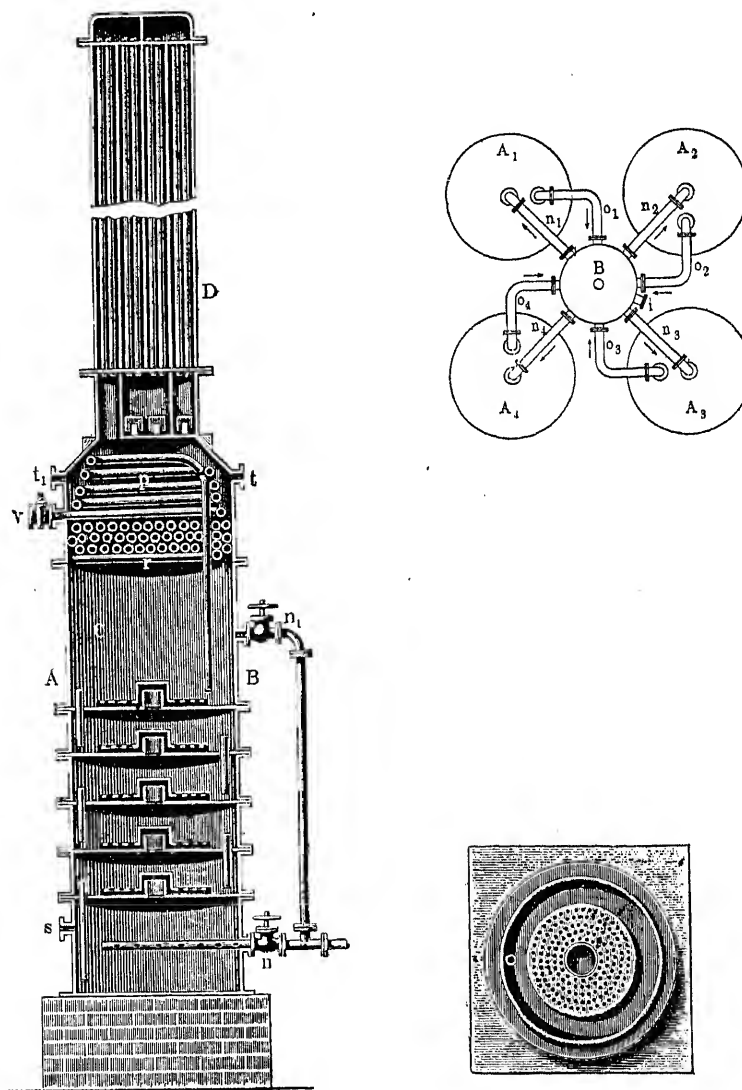


FIG. 168.—FASSBENDER DISTILLING COLUMN.

pipes and for feeding the others with steam in rotation. The fourth view indicates the rotation method of operation as applying to four boilers.

Fig. 168 shows a distilling column employed by Fassbender for the distillation of ammonium bicarbonate. The bottom portion exhibits the characteristics of many

well-known stills, and explanation is unnecessary. The multitubular cooler D is a continuation of the distilling column. A variety of forms are applicable for this purpose, provided they afford the proper essentials in the matter of gas and liquor velocities, cooling surface, etc.

It is known that if the distilling apparatus is properly designed and carefully operated, the ammonia gas is practically denuded of all moisture, and the ammoniacal brine need not be fortified by the addition of solid salt. The stills and apparatus described have been in use at ammonia-soda works for more than twenty years.

Fig. 169 shows a reservoir for mother liquor. It consists of a closed cylinder, placed at some distance above the ground, the mother liquor from the filters being forced into it by compressed lime-kiln gas. The manhole *n* serves for the introduction of the mother liquor. *t*₁ affords access to the flange *t*₁ in fig. 168 by means of a pipe, so that the pressure in both apparatus is in equilibrium, and the reservoir is susceptible to the same pressure variations as the still. The outlet *w* communicates by a pipe with the branch *w* of the coil of the distilling column, fig. 168. Gauge cocks and a

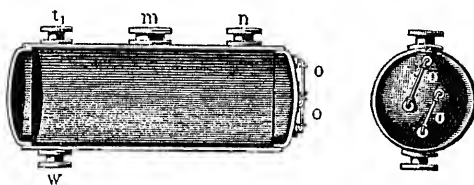


FIG. 169.—MOTHER LIQUOR RESERVOIR FOR REGULATING LIQUOR SUPPLY.

manhole are provided. The bottom of this tank is at least 13 ft. above the inlet *w* of the column. As the diameter of the tank is about 8 ft., any variations of the depth of liquor within cause only a slight difference in the rate of feed to the column. This is a distinct advantage, ensuring, as it does, regularity of working.

The question of steam consumption has frequently arisen, particularly in connection with continental works, where fuel supplies were more expensive and not so readily available as they are in this country. Moreover, the desirability of distilling large quantities of ammoniacal mother liquor in one unit of plant as distinct from the supply of small quantities of liquor to several plants has been realised.

Fig. 170 shows an up-to-date distilling unit, of the Mallet type, which speaks largely for itself. In connection with plants of this type exhaust steam can be used with advantage. One of these columns about 37.5 metres high and 4 metres diameter is capable of dealing with 11,000 gallons of ammoniacal mother liquor per hour having the analysis shown on p. 293. Most of the works having stills of the latter type reduce the ammonia content to 0.01 per cent of NH_3 .

As illustrating the rapidity with which the ammonia is dispersed after the introduction of the limed liquor to the second column, the following figures, given by Bradburn, in dealing with the working of a Solvay column, are peculiarly interesting :

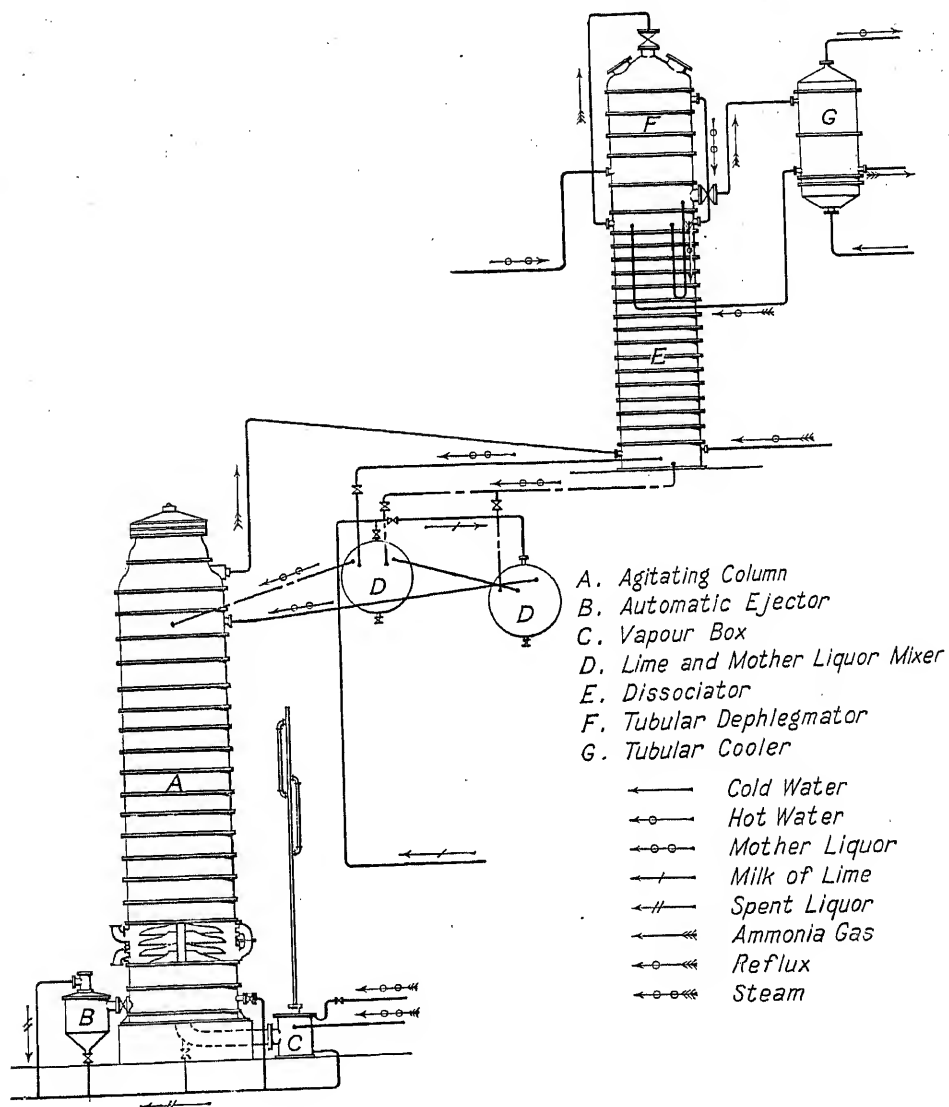


FIG. 170.—MALLET AGITATING COLUMN AND ACCESSORY PLANT.

TABLE 70

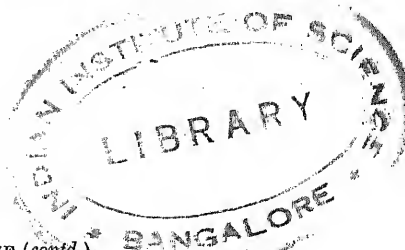
	Grams per Litre.			
	Total Ammonia.	Free Ammonia.	Combined Ammonia.	Carbonic Acid.
Ammoniacal mother liquor . . .	67.2	15.0	52.2	39.3
Compartment No. 21	83.4	39.0	44.4	26.2
" " 20	74.0	30.7	43.3	9.0
" " 19	66.0	23.1	42.9	3.8
" " 18	62.8	20.4	42.4	2.0
" " 17	60.5	18.5	42.0	1.2
" " 16	57.7	16.2	41.5	0.71
" " 15	55.0	13.8	41.2	0.38
" " 14	53.0	12.1	40.9	0.20
" " 13	52.6	12.1	40.5	..
(Limed liquor introduced at this stage.)				
" " 12	39.0	39.0
" " 11	9.7	9.7
" " 10	4.0	4.0
" " 9	1.9	1.9
" " 8	1.0	1.0
" " 7	0.6	0.6
" " 6	0.33	0.33
" " 5	0.20	0.20
" " 4	0.13	0.13
" " 3	0.05	0.05
" " 2	0.03	0.03
" " 1	0.017	0.017

What impresses one about the latest type of still used in the ammonia-soda process is its formidable size and the large volume of ammoniacal mother liquor of high ammonia content distilled per hour, as also the extent to which the ammonia is eliminated from the spent liquor. Moreover, an object lesson is afforded in the employment of exhaust steam, which materially reduces the cost of distillation from a fuel point of view.

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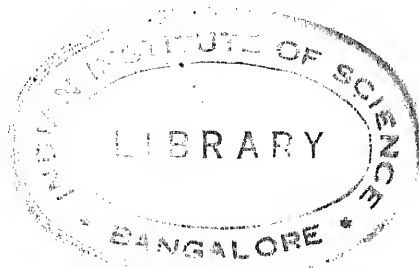
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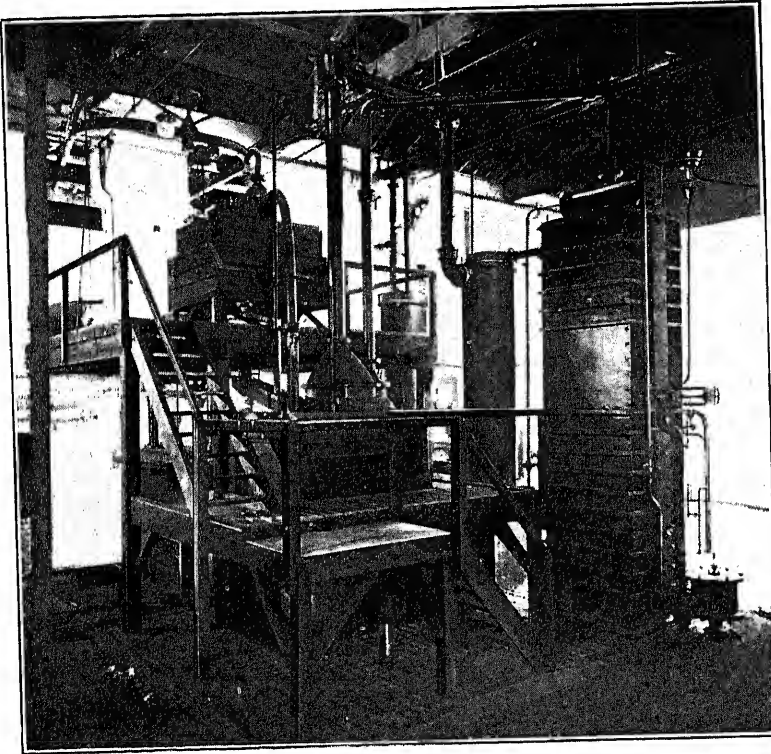


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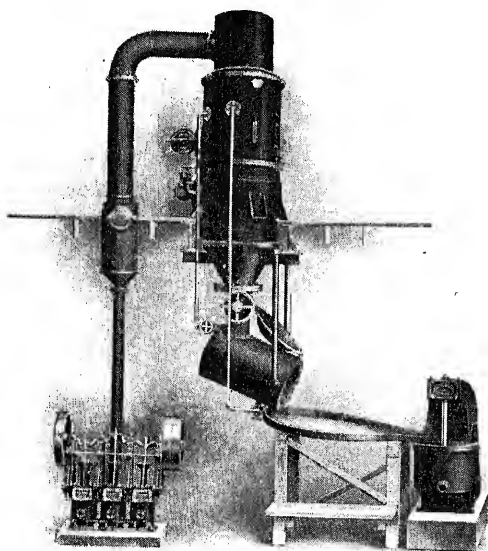
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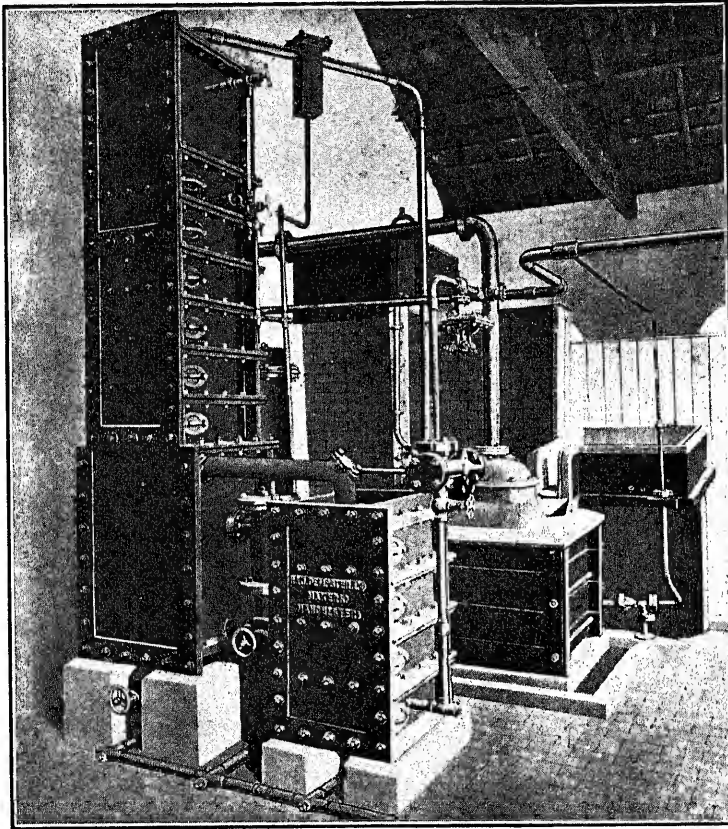
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